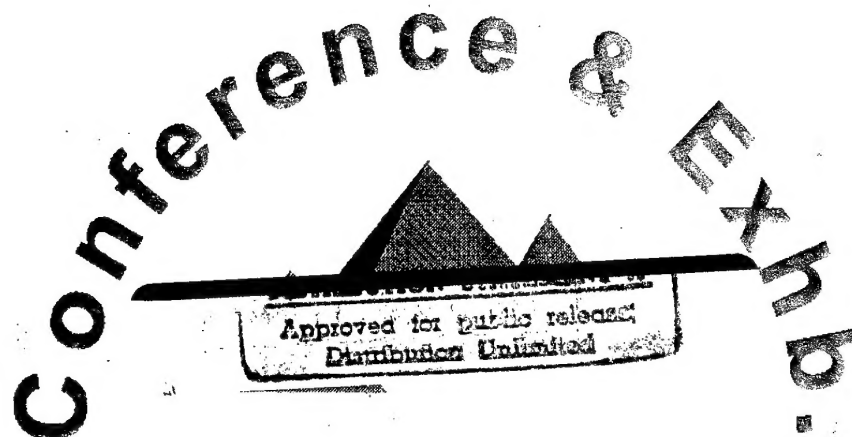


ABSTRACTS

Fourth International Conference on Frontiers of Polymers and Advanced Materials



Jan. 4-9, 1997



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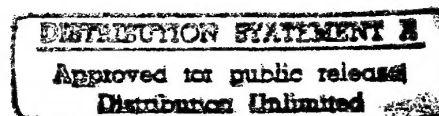


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Plenary Lectures

Light and New Chemistry Expectations for Light Technology

Yoshikazu Ito

Senior Advisor and Senior Counselor, Toray Industries, Inc., Japan

Sunlight has been providing the infinite resource of clean energy constantly since the mythical age, and almost all livings in natural environment have been benefiting from the sunlight through photochemical and biochemical processes for their lives. So, photochemistry is basically an eco-and human- friendly technology.

Fifty years ago, the author found a new photochemical manufacturing process of ϵ -caprolactum, the raw material of Nylon 6, and the process was industrialized on a large scale. But generally the application of photochemical process to mass production of commodity chemicals has been strictly limited owing to the economical reasons instead of ecological aspects, and the photochemical processes has flowered mainly in the field of electronic materials. The typical examples are photo-sensitive materials or photo-resists which support IC industry as one of the most important technologies.

The photochemical process is tried to apply in new fields such as PHB, non-linear optics, and artificial photosynthesis recently. The author expects that the photochemical process will be more widely applied in electro-photonics and bio-photonics in the near future, and believes that photochemistry or light technology will be one of the most promising key technologies in the 21st century.

*Plenary Session [II]
9:00-9:30, Jan. 6*

**New Ways of Scientific Cooperation and Technology
Transfer in Chemistry, Environment and High
Technology and New Materials
PRORAMME OF THE INTERNATIONAL CENTER FOR
SCIENCE AND HIGH TECHNOLOGY, ICS-UNIDO:**

S. Miertus

*International Center for Science and High Technology
Via Grignano, 9, 34014 Trieste, Italy*

Since its establishment in 1988, the International Centre for Science and High Technology (ICS) and autonomous body of the United Nations Industrial Development Organization (UNIDO) has directed its efforts towards the following three scientific fields: *Pure and applied chemistry; earth, environmental and marine sciences and technologies; high technology and new materials.*

In a further effort to focus its action, in 1995 ICS selected the interdisciplinary and at the same time unifying theme of *sound science and high technologies to the environment* in the context of promoting sustainable industrial development as defined and pursued by UNIDO.

In the implementation of the ICS mandate, an important role is played by the following methods of intervention:

- action-oriented research;
- high-level scientific Seminars;
- training courses for technologists from developing countries;
- creation of *Focal Points* and R&D Centres in the developing regions;
- study tours;
- fellowships;
- consultancy and advisory services;
- documentary services through the creation of a Documentation Center (*Clearing-house*)

In the area of PURE AND APPLIED CHEMISTRY THE FOLLOWING sub-programmes will be described in details:

1. *Catalysis in Chemistry and Petrochemical Industry*, with the aim to improve and renew processes and products, namely catalytic processes, as the keystone for efficient conversion into more valuable and cleaner chemical outputs, and for pollution prevention.
2. *Polymeric Materials*, focusing on biodegradable plastics: due to the characteristics of these materials, their use has led to the accumulation of waste which is difficult to recycle, thus leading an environmental problem that could be partially solved by the use of biodegradable plastics.

3. *Bioremediation*, focusing on problems of chemically contaminated solid.
4. *Combinatorial Chemistry and Structure-Based Molecular Design*, focusing on new ways of drug design and techniques.

In this presentation, more details will be given on the sub-programme of *Polymeric Materials, Biodegradable Plastics*.

The ways of cooperation of ICS-UNIDO with developing countries and emerging economies will also be shortly overviewed.

**Materials
for Electronics
and Photonics**

Polymer Blends in Light Emitting Diodes

F.E. Karasz

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Multi-component blends of electro-optically active polymers have been used to enhance brightness and efficiency in, and to simplify construction of, polymer light emitting diodes (PLEDs). By appropriate selection of both spectroscopic properties and phase behavior a PED blend can be designed in which energy migration to the lower band gap component and, if desired, color tuning of the output of the latter can be effected. Of equal utility is the possibility of using the controlled phase separation of two or more immiscible polymers to provide a micro-confinement of excitons in discrete domains of the emitting component. Examples of these concepts will be given.

*Symposium [A]
13:30-15:30, Jan. 5*

Efficient Fullcolour Light Emitting Devices With Polyphenyls

G.Leising, F.Meghdadi, S.Tasch, C.Brandstätter,
W.Graupner, and G.Kranzelbinder

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Soluble and stable polyparaphenylene-type ladder polymers with an extraordinary high degree of intrachain order and exceptionally low concentration of defects in the electronic structure belong to the class of best defined conjugated low-dimensional organic semiconductors currently available. Parahexaphenyl, the highly stable oligomer of polyparaphenylene, can be synthesized in the form of single crystals, disordered thin film and highly ordered epitaxially grown thin films. We present and discuss results on the optical and optoelectronic properties of these polyparaphenylene systems.

We demonstrate the fabrication and characterization of highly efficient red-green-blue (RGB) and white light emitting devices with these electro-active materials. This RGB-devices are fabricated based on a new technique, which allow the realization of fullcolour flat panel displays. The brightness of such RGB emitting devices is higher than 1000 cd/m^2 (for the green device), comparable to that of fluorescent tubes. Using this new technique, we able to produce devices having any desired emission color in the visible and near infrared spectrum.

A high photoluminescence quantum yield and a good environmental stability of the active oligomer or polymer materials, besides many other parameters, are very important to achieve competitive operation lifetimes of these new organic electroluminescence devices.

Photoexcitation experiments on our polyparaphenylenes reveal their high photoluminescence quantum yield and the perspective for being true candidates as solid-state polymer-laser materials due to their pronounced signature or blue-green stimulated emission. We present blue-green laser action in our polyparaphenylene-type ladder polymer.

*Symposium [A]
13:30-15:30, Jan. 5*

Novel Organic Nanostructures and Composites for Light Emitting Diodes

Zakya H. Kafafi, Hideyuki Murata, Darius J. Fatemi and Charles D. Merritt
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Organic light-emitting diodes incorporated in small or large, rigid, flexible, bright and efficient displays will play an important role in the electronic display technology of the y. applications range from hand-held or head-mounded displays to large flat panel screens that can be rolled up or hung flat on a wall. In this talk we review our recent progress at NRL in developing highly efficient, molecular organic light-emitting diodes (MOLEDs). Composites of highly fluorescent organic molecules dispersed in an electron or hole transport organic host, were prepared using high vacuum deposition techniques. MOLEDs based on a one-, two- and three-layered organic nanostructures where the emitting layer serves the dual role of an electron or hole transport were fabricated. High electroluminescence quantum yields (photons/electrons) and luminous power efficiencies (lumens/watt) were realized.

*Symposium [A]
13:30-15:30, Jan. 5*

Pyridine-Based Polymer dc and ac Light-Emitting Devices

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D.K.Fu,** T.M.Swager,** and A.G.MacDiarmid**

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Pyridine-based conjugated polymers and copolymers have been shown to be excellent candidates for polymer light-emitting devices in various device configurations. The high electron affinity of pyridine based polymers enables the use of relatively stable metals as electron injecting contacts. Taking advantages of the better electron transport properties of the pyridine-containing polymers, we fabricate bilayer devices utilizing poly(9-vinyl carbazole) (PVK) as a hole transporting/electron blocking layer, which improve the device efficiency and brightness significantly due to the charge confinement and exciplex emission at the PVK/emitting polymer interface. The incorporation of a conducting polyaniline network electrode into PVK reduces the device turn on voltage significantly while maintaining the high quantum efficiency, thus improving the device power efficiency. Through sandwiching an emissive polymer layer between two redox polymer layers we create symmetrically configured alternating current light-emitting (SCALE) devices that operate in both forward and reverse modes, as well as ac mode. Among advantages of SCALE devices are ac circuitry, use of stable high work function contact metals such as gold, and reduced electrochemical degradation.

This work was supported in part by the Office of Naval Research.

*Symposium [B]
16:00-17:50, Jan. 5*

Electronic Structure of Luminescent Conjugated Polymers: Relevance to Polymer-Based Light-Emitting Diodes

J.L.Brédas, D.Beljonne, J.Cornil, R.Lazzaroni, and D.A.dos Santos

Service de Chimie des Matériaux Nouveaux

Centre de Recherche en Electronique et Photonique Moléculaires

Université de Mons-Hainaut

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The tremendous progress currently achieved in luminescence efficiency, color control, and lifetime of conjugated polymer-based light-emitting diodes, has made such devices increasingly attractive for commercial applications.

In this contribution, we review the basic ingredients of the electronic structure of conjugated polymers. We then emphasize those characteristics that are directly related to the luminescence properties:

- nature of the lowest-lying single and triplet excitations; we pay special attention to the evolution of the state energies with chain length and the ways to prevent at best the inter-system crossing process;
- influence of inter-chain interactions; of special importance is the possibility of establishing the parameters that would favor either intra-chain excitations (which are relevant to electroluminescent devices) or inter-chain charge-separated excitations (which are relevant in the context of photovoltaic devices)
- chemical structure of the interfaces formed between conjugated polymers or oligomers and metals.

*Symposium [B]
16:00-17:50, Jan. 5*

Electrical Transport and Magnetoconductivity in Doped Polypyrrole

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Polypyrrole doped with Toluene-p-sulfonate [PPy(TOS)] has been prepared electrochemically by using different current densities. It was found that, electrical conductivity (σ) has a great dependence on the current densities i.e, (~ 0.17 and 50 S/cm at $j=0.5$ and 7.5 mA/cm² respectively). Conductivity has been fitted according to Mott's equation in a wide range of temperature from 18 K to 200 K with a good agreement. In addition, it was observed that, polypyrrole samples have a strong positive magnetoresistance at very low temperatures and it has a quadratic behavior at small magnetic fields, up to $H=5$ T.

*Symposium [B]
16:00-17:50, Jan. 5*

Side Group Thermotropic Liquid Crystal Polymers

Jean-claude Dubois

Thomson-Csf.LCR.Orsay, France

This paper would review some aspects of the structures and electrooptical properties of side chain liquid crystal polymers. Side chain liquid crystal polymers are formed by attaching rigid units to a flexible chain. Decoupling of the side group by using a flexible spacer allows the main chain motion to give the anisotropic arrangement of the side chain and the polymer may exhibit liquid crystal properties. So side group (or side chain) LCP are reminiscent of low molecular weight LC and their properties are characterized by a combination of LC specific and polymer specific properties. The LC specific properties include the formation of any of the different types of mesophases.

From the different synthetic methods one can propose polymers exhibiting nematic and smectic mesophases. If the side group and/or the chain are chiral one can obtain cholesteric phase or chiral smectic C phase (Sc*). This can be achieved also by mixing a chiral compound with the SCLCP. Sc* SCLCO are of particular interest and their behavior is described in the 1st part of this conference. Many reviews [1,2,3] of SGLCP describe that the structure of the backbone (main chain) the spacer (flexible linkage) and the side group (mesogenic unit of the SCLCP) has been widely varied. For example the most described backbones include poly(acrylates) or poly(methacrylates), poly(siloxanes) and poly(phosphazenes). Typical spacer groups consist of 3 to 12 methylene units. The pendant rigid groups are chosen among those which constitute the LMWLC's including biphenyl, phenyl benzoates and benzodaniline. Combinations of a rigid main chain polymer and a lateral side group have been also described. Different structures and their mesophases will be given in the first part of the talk.

Orientation by an electric field has been studied and allows the realization of polymer displays, this will be described in the second part of this talk.

A polymer specific property of importance in SCLCP is the formation of glass on cooling because of partial decoupling of the flexible backbone motion from the side chain. The glass transition temperature T_g is determined by the chemical constitution of the backbone and in some cases the LC order may be frozen in to yield an anisotropic glass with unique optical properties. Specially in the field of non linear optics active side group can be attached, oriented and frozen under electric field, these important electrooptical properties are described in the last part of this conference.

*Symposium [C]
10:30-12:30, Jan. 6*

Advanced Liquid Crystal Optical Shutters For Display Devices And Spatial Light Modulators

Tadahiro Asada

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Polymer Cell-Wall Type Liquid Crystal (PCW-LC) light shutter has been developed. This is a kind of liquid crystal-polymer complexes (LCPCs), utilizing the light scattering of cholesteric focal conic textures. However, its abilities are superior to other LCPCs reported. For examples, the driving voltage is very low (Ca.5-6V) and the contrast ratio is very good (more than 150). As the cholesteric liquid crystal grains are covered with thin polymer films, the polymer content is very small, around 10 wt%. This type of LC light shutter has the great advantage of non-polarizer usage. Therefore, this type of light shutter can be useful for not only liquid crystal display devices but also other applications, for example, spatial light modulator (SLM). In this work, PCW-LC is introduced at first, and then the applications of PCW-LC light shutter to SLM is described. Non-polarizer usage makes it easier to combine it with the other component parts for producing a SLM. The reduction of components parts makes more effective SLM, because of minimizing light energy loss by puzzled light originating from the binding surfaces. We intend to develop this type SLM, because it has the great advantage in making without pixelization.

*Symposium [C]
10:30-12:30, Jan. 6*

Liquid Crystal Photorefractivity for recording holographic gratings

I. C. Khoo, B. D. Guenther, Michael Wood and P. Chen

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University Park, PA 16802 USA*

We present a detailed discussion of the fundamental mechanisms for recently observed persistent photorefractive effects in nematic liquid crystal films [I. C. Khoo, Optics Letts.20, 2137 (1995); IEEE J. quant. Electronics JQE ;2, pp525-534, 1996]] . In dye- or Fullerene C₆₀ -doped films, it is observed that the induced director axis and refractive index grating could persist for a long time. Furthermore, these stored grating can be electrically switched on and off at ms-subms speed. Our recent experiments have shown that these effects could also be observed with near W or near IR lasers, in planar aligned as well as homeotropic nematic liquid crystal films. Furthermore, the application of a ac field has been shown to enhance the grating formation efficiency. A theoretical analysis of the various space charge fields, dielectric torques, and estimates of their magnitudes and critical assessment of their roles in the observed holographic grating formation process will be presented.

Molecular Design and Properties of Novel Chain Liquid Crystal polymers for NLO Applications

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For a wide variety of $\chi^{(2)}$ applications, namely frequency doubling, electro-optic modulation, frequency conversion and photorefractive effect, noncentrosymmetric media are required. Among others, composite polymeric materials seem to be promising candidates for these future practical applications because of their great diversity, high damage threshold limits and ease of processing into high optical quality thin films. They are also characterized by an ultrafast THz response time. In these materials, however, intrinsic polar order is absent. A static electric field or an all optical poling process must be employed in order to impart an overall polar orientation of the active chromophores. For poled polymers, the macroscopic second order nonlinear optical (NLO) susceptibility, $\chi^{(2)}$ is given by the well-known relation:

$$\chi^{(2)}(-\omega_3; \omega_1, \omega_2) = NF \langle \beta(-\omega_3; \omega_1, \omega_2) \rangle$$

where N is the number density of the NLO molecular species, F is the local field factor and β is the molecular first hyperpolarizability. The brackets denote an averaging over all molecular orientations. To achieve maximum values for $\chi^{(2)}$, the challenge then is to optimize not only the magnitude of the molecular hyperpolarizability and the number density but also the alignment of those species responsible for the NLO response. Liquid crystals offer a way to exploit the intrinsic axial ordering of a mesophase for enhancement of polar ordering. Recently it has been shown theoretically and experimentally that, depending on the degree of axial ordering, the performance may be improved by a factor of 1 to 5 by using liquid crystal polymers (LCP's) instead of amorphous isotropic polymers.

In this paper, we will present a new approach for the design of side chain LCP's with large and stable second-order NLO properties. Emphasis will be put on polymaleimide derivatives with high glass transition temperatures. Real time *in situ* second Harmonic Generation experiments will be presented. Special attention will be paid to the influence of axial order on the poling of the NLO pendant groups. The results will be compared to the predictions of molecular statistical models.

*Symposium [C]
10:30-12:30, Jan. 6*

Towards A Two-Dimensional Grid Polymer

Josef Michl

Department of Chemistry and Biochemistry, University of Colorado,
Boulder, CO80309-0215, U.S.A

We report an approach to two-dimensional polymers of 5 - 10 Å thickness and lateral dimensions large on the molecular scale, in the form of a grid with openings of a controlled size in the range from 1 to 30 Å in diameter. The grid consists of connectors carrying rods joined linearly by sturdy chemical bonds, providing enough strength for practical applications such as size-exclusion separation or surface blocking for electrochemical deposition of quantum dots. The grid is synthesized by polymerization of monomeric units shaped like a star or a cross, induced by linear coupling of arm ends. During the polymerization process, the monomer molecules are confined to a two-dimensional surface, preventing the formation of a three-dimensionally cross-linked polymer of the more usual kind. This is accomplished by mounting them covalently on top of pedestals provided with tentacles that contain functional groups with large affinity for the surface used. If desired, the tentacles can be severed by a chemical reaction once they have accomplished their purpose, facilitating the ultimate removal of the polymeric grid from the surface. The adsorption of the pedestal also orients the mounted monomer molecules properly for the desired coupling, with arms parallel to the surface, as evidenced by grazing incidence reflection IR spectroscopy. Translational and rotational mobility required for proper alignment of the arm ends during the coupling reaction is provided by using a liquid surface, mercury. The use of a liquid also provides an opportunity for the transfer of the resulting grid to other substrates, solid or perforated, by Langmuir-Blodgett techniques. Three choices of monomers, based on 1,3,5-trisubstituted benzenes, tetrasubstituted cyclobutadiene cobalt complexes, and *meso*-tetrasubstituted porphyrins will be described. Issues such as possible annealing of the initially formed regular domains into larger regular domains through the use of reversible coupling reactions, and minimization of the number of defects, will be discussed. At present, we believe to have surface IR and resonance Raman spectroscopic evidence that we have produced the first grids of this kind, and we are attempting to obtain information about their long-range order, i.e., to find out whether they are two-dimensionally crystalline or amorphous in nature. In the future, we foresee a large number of variations on this theme, with the two sides of the grid differentially functionalized, with thicker aperiodic covalent multilayer structures built one layer at a time, and with a variety of static or mobile active groups, such as electron donors and acceptors, light absorbers and emitters, non-linearly optically active units, charged or magnetic units, etc.

Acknowledgement. This work was supported by the U.S. National Science Foundation and Department of Energy.

*Symposium [D]
13:35-15:45, Jan. 6*

A Model for Single-Molecule Information Storage

**Christopher B. Gorman, Brandon L. Parkhurst, Wendy Y. Su,
Kang-Yi Chen, Igor Touzov**

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We are constructing a model system for the storage of information on a surface using individual molecules. Several demonstrations must be made to prove the feasibility of such a system using scanning probe microscopy (e.g. STM/AFM or a combination of both). The probe tip must be able to site-specifically control the adsorption/desorption of individual molecules or small, isolated collections of molecules on a flat surface. The probe tip must be able to switch the molecule(s) between two stable states that are distinguishable when the tip is used in a read mode. We have synthesized a class of new electroactive molecules and are elucidating tip-induced chemical reactions on well-defined organic surfaces that should have utility in assembly and addressing schemes.

Although a number of molecules which are "switchable" have been discussed in the literature, isolation of information, assembly, and addressing of individual molecules have received much less attention. A multifunctional molecule will be necessary if it is, for example, going to be a redox switch and simultaneously have a tailored interaction with a surface. We are pursuing this scheme using an encapsulated electroactive unit. Our model system consists of an inorganic cluster surrounded by dendrimeric ligands which will act as an organic coating. The electroactive core will permit the use of the molecule as a redox switch. The organic coating will be used to isolate the information held by the redox state of the core. Placement of specific functional groups at periphery of the dendrimeric ligands may be used to control the interaction of the molecule with the surface.

*Symposium [D]
13:35-15:45, Jan. 6*

Shape And Surface Coverage Dependence of The Catalytic And Dynamic Properties Of Nanoparticle

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An important characteristic of nanoparticles is the sensitivity of their properties to the type of surface atoms and their bonding properties. We shall demonstrate this in two important properties of metallic and semiconductor nanoparticles. Recently, we have shown that the shape of platinum nanoparticles can be controlled by changing the concentration ratio of the capping polymer to that of Pt^{2+} ions in aqueous solution. Since catalysis depends greatly on the extent of the valence unsaturation of the surface atoms, one expects that it should be sensitive to the shape of the nanoparticle. Results will be presented to show that while the tetrahedral particles we used are $2/3$ the size of the cubic ones, they are 400% more efficient in catalyzing the electron transfer reaction between thiosulfate and hexacyanoferrate III. The reason for this will be discussed.

In CdS nanoparticles, the dynamics of the trapping of the electron-hole pair greatly changes if its surface is covered by a shell of HgS, as present in the CdS-HgS-CdS quantum dot-quantum well (QDQW) nanoparticles. Using femto second spectroscopy, we have shown that the trapping sites change from being surface CdS molecules in CdS nanoparticles to being the HgS molecules in the quantum well of the QDQWnanoparticle.

*Symposium [E]
16:00-18:00, Jan. 6*

The Role of London Forces in Defining Order in Poled and Self-Assembling Dipolar Materials

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The precise organization of nanoscale molecular structures is, in general, defined by competing forces although frequently one force will clearly dominate the architectural definition. In the present communication, we consider nanoscale order which results when external forces are used to introduce eccentric order in competition with centric order inducing intermolecular electrostatic interactions and entropic (or thermal) effects which attempt to induce random order. Detailed comparison between theoretical results and results obtained for electric field poling (as the external ordering force are presented for a number of molecular systems spanning a range of electrostatic interactions. The starting point for the theoretical analysis is the consideration of Debye-Fowler/London theory where restrictive approximations on the shape of molecules are avoided. Following London, we explicitly include the interactions (permanent dipole, induced dipole, and dispersion) depending on the variables molecular dipole moment, μ , polarizability, α , and ionization potential. Calculations are modified to include effects of molecular dynamics which are necessary to account for certain non-equilibrium effects. To facilitate quantitative comparison between theory and experiment, the molecular systems were chosen to be dipolar chromophores exhibiting easily measured electro-optic effects, birefringence and dielectric effects. In the case of electro-optic experiments, eccentric order is directly measured and theory correctly predicts that for all materials studied maxima in the graphs of electro-optic effect versus chromophore number density will be observed at high number densities (at high chromophore-chromophore electrostatic interactions associated with reduced average separation of chromophores). Indeed, experimental data are simulated with surprising accuracy and without introduction of adjustable parameters. Comparison of theory and experiment permits the identification of two important classes of behavior. In the case where $KT >$ intermolecular electrostatic interaction energies, the only effect of intermolecular electrostatic interactions is to attenuate the effect of the poling field. This can be viewed as an equilibrium effective field which acts to simply attenuate the action of the external poling field. For the case where $KT <$ electrostatic interaction energies, non-transient aggregation is observed which depends strongly on the details of the molecular dynamics. Light scattering is frequently observed for this latter case and occasionally pulsed poling fields (where μE is comparable to intermolecular electrostatic interactions) can be used to disrupt aggregates and improve poling efficiency (acentric order). Theory allows correct prediction of the relationship between chromophore geometric structure and nanoscale order.

Symposium [E]
16:00-18:00, Jan. 6

New Generation Nanostructured Multifunctional Materials for Electronics and Photonics

Paras N. Prasad

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Buffalo, NY 14260-3000*

Multifunctional materials which simultaneously exhibit more than one functionality and new properties derived from a combination of two or more functionalities will play a crucial role for future development of electronics and photonics.

This talk will discuss new generation multifunctional materials developed in our laboratory using a combination of design, chemical synthesis and processing at nanometer scale. Controlled nanoscale synthesis and polymerization in the cavity of a reverse micelle represents a new approach. At bulk level, processing can be used to achieve nanoscale control of domain size to introduce multifunctionality by utilizing each domain to perform a specific function. We have utilized this approach to produce unique materials called multiphasic nanostructured composites by sol-gel processing. Nanoscale synthesis and processing have also allowed us to produce a new class of hybrid inorganic:organic composite materials for photonics and electronics. Specific application of new materials to high density three-dimensional optical data storage will be presented.

The talk will conclude with a 6 minute video presentation of 3-dimensional optical data storage utilizing materials developed in our laboratory. This video will present the world's smallest Bugs Bunny show.

*Symposium [E]
16:00-18:00, Jan. 6*

Optical Nonlinearities in Nanoparticle Composites

D.D. Bhawalkar, K.C. Rustagi

Centre for Advanced Technology, Indore 452 013, India

Linear and nonlinear optical response of nanoparticle composites is influenced by the quantum confinement of electrons, excitons and the dielectric confinement of the electromagnetic waves. The present status of the phenomenological theories describing the nonlinear optical response of a variety of composite materials is reviewed. The materials considered include semiconductor particles in glass and in Langmuir Blodgett Films, colloidal metal nanoparticles and fullerenes doped porous glass.

We analyse the absorption, fluorescence and Raman Spectra in light of the available theories of electrons, phonons and the electron-phonon interaction. Experimental results on Z-scan degenerate four wave mixing and the optical Kerr effect are presented and analysed.

The potential of nonparticulate Langmuir Blodgett Films and fullerene doped porous glass is examined by a careful analysis of their linear optical properties. Results on their nonlinear optical properties will be presented.

*Symposium [F]
10:30-12:40, Jan. 7*

Nano Technology in Microemulsions: Radiolytic Production of Metallic Nanoclusters in A Quaternary System

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Chemistry Group, Bhabha Atomic Research Centre
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The preparation of metallic nanoclusters is of great importance both from the view of technology as well as basic research. The ability of these clusters to transfer electrons readily as compared with bulk materials makes them very valuable in the field of catalysis, photography, optics etc. The size of these clusters places them between atomic and bulk states, making them very useful engineering materials.

Microemulsions have attracted a great deal of interest in recent years. It is possible to carry out very interesting chemistry in the microwater droplets in these systems. The size of these droplets can be in the region of 10 to 20 nanometers (nm) making them ideal for preparing nanoclusters. Here we report the preparation of cadmium and copper nanoclusters in a four component microemulsion using electron pluses to produce metallic aggregates in the water pool.

The technique of pulse radiolysis has been employed to study the kinetics, formation and stability of these micro aggregates. The microemulsion used was composed of cyclohexane/surfactant/water/alcohol. The most unique feature of our system is the absence of complexing agents. The cadmium particles produced show remarkable stability in solution with respect to the presence of air as observed by absorption spectrophotometry. The particle size has been estimated from the near U.V. absorption and is lower than 10 nm. Copper nanoclusters have also been produced in the same system by the pulse radiolysis route. Production of other metallic nanoclusters in microemulsions is also being studied.

We are also studying macromolecular solutions for producing nanoaggregates through the radiolytic route. Gelatin seems to be the most promising of the macromolecules tried.

*Symposium [F]
10:30-12:40, Jan. 7*

From Gas Phase Clusters to Polymers and Nanoparticles: A Route to Novel Materials

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Intracluster cationic polymerization reactions possess a distinct form of chemistry that has not been previously explored. In the gas phase at low pressures, eliminative polymerization may predominate. However, in the same systems at high pressure, the ionic intermediates may be stabilized and addition without elimination may occur. In clusters, our results show evidence for both stabilization by fast evaporation (boiling off) of solvent molecules and for eliminative polymerization. The competition between the condensation reactions and monomer evaporation can control the ultimate size that the polymer can reach in the cluster. Several examples of intracluster polymerization will be discussed.

We will also present results from a novel technique for the synthesis of cationic polymers containing ultrafine metal particles. In the experiments, laser vaporization of metal targets is used to generate ultrafine metal particles and cations which are capable of catalyzing the cationic polymerization of isobutylene. This method can lead to the generation of new polymeric materials with unique properties.

In another study, a method which combines laser vaporization of metal targets with controlled condensation in a diffusion cloud chamber is used to synthesize nanoscale metal oxide, carbide and nitride particles (10 - 20 nm) of homogeneous size and well - defined composition. The microscale structures of the SiO_2 and Al_2O_3 particles exhibit interesting web-like matrices with a significant volume of voids. These aggregates are very porous and have a very large surface area ($500 \text{ m}^2/\text{g}$). Bright blue photoluminescence from the nanoparticle silica has been observed upon irradiation with UV light. The photoluminescence is explained by the presence of intrinsic defects of the type Si(II)° in the amorphous silica. This material could have special applications in optical devices, catalysis and as a polymer reinforcing agent.

*Symposium [F]
10:30-12:40, Jan. 7*

Flexible Single Crystal Semiconductors for Fabrication of Semiconductor/Polymer Structures

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Semiconductor/polymer combinations have been used extensively for various purposes, with polymers being the supporting materials, such as packaging. As the field of photonics with polymer materials advances at a fast pace, it is important to incorporate the two kinds of materials in a more intimate fashion, so as to take full advantage of their desirable features. While polymer materials possess flexibility and low cost, traditional semiconducting (non-polymeric) materials are characterized by high crystal quality and superior electronic and optical properties; these can be further improved by epitaxial growth techniques in the form of nanostructures. A combination of *all* these features will be important for future photonic applications.

Our approach to this problem is to fabricate epitaxially grown semiconductor quantum well structures in flexible forms, which can be incorporated in polymer-based structures. The flexibility of single crystal semiconductor films depends mostly on their dimensions. The maximum strain in a 1 μm thick layer (typical for samples grown by molecular beam epitaxy) with a 1 mm radius of curvature is around 0.01%, which is far less than the lattice mismatch in many semiconductor heterostructures and can be tolerated by almost all material systems. Since all epitaxial structures involve substrates, a lift-off technique has to be used for transferring the epitaxial layer to flexible materials. In this study, we developed a method for transferring epitaxial ZnCdSe/ZnSe quantum well structures to silicone films and flexible indium films. The choice of silicone for our initial study simply comes from its availability in the form of adhesives. The transfer process consists of mechanical polishing and selective chemical etching.

As a means of evaluating the sample quality after processing, photoluminescence (PL), optical reflectivity and transmission spectroscopies were used at room temperature for samples with silicone (because silicone hardens at low temperatures) and at 10 K for samples with indium. Experimental results on the flexible ZnCdSe/ZnSe quantum well structures, grown by molecular beam epitaxy indicate very little degradation. A comparison between flat and curved (to a 1 mm radius of curvature) regions showed very little difference, both in the line shape and in the peak position. It can be concluded that the strain in the curved region is negligible because optical transition energies are very sensitive to strain.

It should be emphasized that the results presented here are not limited to ZnCdSe/ZnSe heterostructures. With a proper selective etching solution, which is the case for most of the semiconductor materials, such a technique can be readily applied to other systems. Experiments using GaAs-based heterostructures are underway. We have also extended our effort to characterizing adhesion and other relevant properties at the interfaces of II-VI semiconductors and polymers.

*Symposium [F]
10:30-12:40, Jan. 7*

Electronic Effect of Frenkel-Wannier-Mott Excitonic States in Organic-Inorganic Semiconductor Quantum Wells

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Organic and inorganic semiconductor quantum wells exhibit new optical properties due to quantum confinement and reduced dimensionality. Recently the development of growth techniques allows the preparation of composite organic-inorganic semiconductor nanostructures. A new type of excitonic states was considered in a heterostructure of a molecular layer and a semiconductor quantum well. This new hybrid excitation can be described as Frenkel-Wannier-Mott exciton. The coupling of the electronic excitation can show large dipole moment and strong non-linear phenomena. These properties are very promising for devices applications in optoelectronic. Frenkel excitons have very strong oscillator strengths while Wannier excitons are very sensitive to external perturbations: static electric and magnetic fields[2]. Our interest is centred on the mixed exciton formation, calculations was performed for the system composed by a monolayer of organic semiconductor (anthracene) weakly adsorbed at a single quantum well of inorganique semiconductor (ZnSe/ZnCdSe). The hybrid Frenkel-Wannier-Mott excitonic states may be characterized by the relatively enhanced oscillator strength (typical for Frenkel excitons) and the large radius (Wannier-Mott excitons). The effect of the static electric field is discussed.

*Symposium [F]
10:30-12:40, Jan. 7*

Application of Thin Films of Conjugated Polymers in Sensors for Hydrocarbon Vapors, Liquid Crystal "Light Valves", Microcontact Printing and Electrochemical Light Emitting Devices

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Although thin films of conjugated polymers on substrates are well known they have only previously been extensively used in LED devices. We here report their use in the fabrication of several different types of devices and in microcontact printing. Thin films of polypyrrole, polyaniline and polyaniline tetramer when deposited on interdigitated gold electrodes serve as excellent sensors for hydrocarbon vapors. Rapid and reversible changes in resistance are observed on alternate exposure to, e.g., toluene vapor and nitrogen respectively. Computer derived hydrophobic films are produced on hydrophilic glass substrates by a microcontact "stamp" printing technique. A hydrophobic pattern is produced when the silicone resin stamp ("inked" with $C_{18}H_{37}SiCl_3$) contacts the hydrophilic surface. Thin films of polypyrrole and polyaniline are deposited preferentially on the hydrophobic pattern from dilute aqueous solutions of the polymerizing monomer. Features having 10-20 μm dimensions can be produced. Longer exposure to the polymerizing pyrrole solution also results in deposition on the untreated hydrophilic surfaces. The Vis/UV spectra of the polypyrrole suggests a tight coil molecular conformation for the film deposited on the hydrophilic surfaces and an expanded coil conformation for the film deposited on the $C_{18}H_{37}$ -hydrophobic surfaces. Films of metals such as nickel are deposited selectively by an electrodeless process on the hydrophilic patterned substrate surfaces. When the patterned polypyrrole is used as one electrode and ITO conducting glass is used as the other electrode, patterned polymer dispersed liquid crystal displays are readily produced. Novel flexible, liquid crystal light valves using polymer dispersed liquid crystal constructions have been fabricated using thin films of conductive polypyrrole on flexible transparent substrates for both electrodes. Characteristics of light-emitting *electrochemical* devices M/MEH-PPV+TBATS/ITO (where M=Al, Cu, Au and TBATS=tetrabutylammonium p-toluenesulfonate) are reported. Light emission is observed in forward and reverse bias modes in each device.

Acknowledgements: Supported in part by the University of Pennsylvania Materials Research Laboratory (NSF Grant No. DMR-91-20668), the Office of Naval Research and by a dual use Tech-nology Reinvestment Program, Contract No. N00014-95-2-0008.

*Symposium [G]
16:00-18:00, Jan. 7*

Organic and Inorganic Photorefractive Materials

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The photorefractive effect as space-charge induced refractive index changes in inorganic electro-optic crystals and ceramics has been investigated extensively during almost three decades. In organic crystals the same effect had been discovered in COANP:TCNQ in 1989. Later on, three other organic crystals prepared in our laboratory have been shown to be photorefractive. In addition to the photorefractive effect a similar effect due to the formation of space-charge induced orientational gratings has been found in poled polymers by several authors. Since the origin of the refractive index changes had not been recognized properly at the beginning, this effect was also called "photorefractive".

A lot of progress has been made both in the development of organic and inorganic "photorefractive" materials in recent years. We will summarize some of these developments and compare the figures of merits for both groups of materials for applications in optical parallel processing, data storage and guided-wave devices.

*Symposium [G]
16:00-18:00, Jan. 7*

Photorefractive and Light Emitting Polymers for Optical Applications

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Recent advances in the development of highly efficient photorefractive polymers will be presented. The performance of these new materials has been improved by orders of magnitude in the past years. We have now developed photorefractive polymers for wavelength ranges of 630-830 nm. Our recent progress in IR-sensitive photorefractivity will be discussed. Examples of applications including optical correlations for security verification will be presented. We will also present our latest results in the development of organic light emitting diodes.

*Symposium [G]
16:00-18:00, Jan. 7*

Progress Towards WDM Demultiplexing with Sum and Difference Frequency Generation in Poled Polymers

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Wavelength multiplexing, that is the simultaneous use of multiple wavelengths to send communications signals, requires a novel device for separating the signals into separate information channels at the receiving end. It is also frequently advantageous to shift the center wavelength of all channels. Normally these two functions are implemented in separate devices. Our approach is to perform both in a single device using second order nonlinear interactions in poled polymers. We plan to utilize sum frequency generation by two oppositely propagating guided waves in a poled polymer to spatially separate the different wavelengths. Difference frequency mixing in a parallel array of channel waveguide with a reference laser then yields the signals of interest in different channels.

This device requires that both frequency conversion processes be performed as efficiently as possible. As a prelude to difference frequency generation, we have developed second harmonic generation by co-propagating beams with 1550 nm input using a variety of phase-matching geometries to produce figures of merit approaching those of LiNbO_3 , namely $15\%/W\text{-cm}^2$. Using modal phase-matching, we achieved phase-matching over 8 nm in channel waveguides using the DANS polymer. These experiments will be described, as well as the materials properties needed for further progress.

Progress has also been made in sum frequency generation by counter-propagating beams. Orders of magnitude improvement in the efficiency were achieved by using multilayer techniques for quasi-phase-matching. To date, the efficiencies are superior to those in other materials systems. Again we describe the progress, and the material properties needed for the next generation of devices.

*Symposium [H]
10:30-12:30, Jan. 8*

Waveguide Devices by Organo-Photonic Materials

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Organic materials for photonic devices are very important in photonic interactive communication system of the world.

We are researching practical applications of photonic devices by organo-photonic materials with waveguide type including optical fiber. In this talk (1) dye doped optical polymer fiber amplifier with graded index which exhibits very high gain amplification up to 30 dB for pulse operation is presented. (2) Also we observed generation of solitary wave with 100 fs pulse duration in organic nonlinear optical thin film waveguide with about 5 mm device length.

*Symposium [H]
10:30-12:30, Jan. 8*

High-Speed Photonics Polymer --Transmission and Display--

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Author describes two main topics supporting multimedia society. One is high-speed polymer optical fiber (POF) for data communication. The other is the novel brighter liquid crystal display (LCD) backlight.

We have proposed a high bandwidth graded-index polymer optical fiber (GI POF), and have demonstrated several giga bit per second (Gbit/s) transmission in the 100 m GI POF link. However, the transmission distance was limited to 100 m due to the intrinsic absorption loss. A new GI POF with low attenuation even at 0.5-1.3-mm wavelength region is presented in this paper. The perfluorinated polymer base GI POF has many advantages; one is no serious absorption peak in the range of 0.6-1.3-mm wavelength in its attenuation spectrum, and the attenuation at 1.3 mm where the LD for silica fiber operates is about 50 dB/km. Another is the low material dispersion compared with PMMA and silica, which allows more than 10 Gb/s transmission in 1 km link. In addition, no modal noise in the GI POF link is observed. Since the PF polymer is very flexible and stable for many chemicals and is not flammable, outdoor use of the PF polymer base GI POF will be possible as well as silica fiber. Therefore, we believe that these experimental and theoretical aspects suggest that the PF polymer base GI POF will be one of the promising candidates for "the last one mile".

We have also proposed a new material for light source medium, which is the highly scattering optical-transmission (HSOT) polymer. This polymer material is made of PMMA resin and contains a small amount of other resin consisting of specified microscopic heterogeneous structure inside the polymer. Therefore, the incident beam is efficiently converted to the homogeneous illumination from the surface of the HSOT polymer by multiple scattering due to such heterogeneous structures. This HSOT polymer was applied to the LCD backlight with 8.4 inches which has two times brightness of the conventional transparent PMMA-based backlight system in spite of much simpler system.

*Symposium [H]
10:30-12:30, Jan. 8*

High Density Disc Storage by Multiplexed Microholograms

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High capacity storage systems with fast data access are essential prerequisites for future multimedia services. For many applications, optical memories such as compact Discs (CD) are preferred to magnetic high density systems due to the low production costs and high portability. Systems that use optical discs as storage media are highly developed and already commercially available on a large scale. The recently developed Digital Versatile Disc (DVD-ROM), which provides a storage capacity of 4.7 GByte, allows applications such as playback of 135 minutes of movie film using data compression with the MPEG II standard. Although rewritable materials (in particular phase change materials) have been subject to extensive research for some time, DVD-WORM or DVD-RAM solutions are still not available in the marketplace. A further increase in the storage capacities of these systems is expected when blue laser diodes become commercially available and dual layer systems are developed. The above mentioned optical disc systems store the data two dimensionally on the surface only. On the other hand, three dimensional storage systems have been proposed on the basis of holographic recording techniques. These systems use either photorefractive crystal cubes or layers of photopolymers to record simultaneously complete digital data pages. Experiments demonstrate that holographic methods allows high storage capacities and very high data transfer rates but they need complex write and read-out devices, a fact which has up to now impeded the design of commercial products.

In contrast to the formerly discussed techniques, we investigate a holographic memory that combines the advantages of both the bitwise storage from existing disc systems and the three dimensionality known from holographic approaches. For this, an optical disc is coated with a photosensitive material and, rather than storing the information by data pits on the surface, volumetric microholograms are induced to vary locally the reflectivity of the disc. In order to create the microholograms, a laser beam is focused within the sensitive layer. A reflecting unit underneath the disc reflects the beam and thereby a standing waver in the region where the incoming and the reflected beam overlap is created. Thus a Bragg grating is inscribed into the material. Photopolymers are extremely promising storage materials since they are highly transparent and undergo faintly large index changes upon light exposure.

Moreover, these microholograms are strongly confined and therefore can be arranged very close to one another. By means of holographic multiplexing techniques a certain number of micro holograms, each characterized by a distinct wavelength and angle, can be stored at the same location in the photopolymer. This gives rise to very high storage densities. For a disc the size of a CD, storage capacities in excess of 40 GByte per disc and data transfer rates of about 50 Mbit/s are estimated.

The Role of Molecular Design in The Preparation of Electronic Materials

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The concepts of molecular design have become well accepted in molecular biology. The idea that researchers can reduce time spent in the laboratory by judicious pre-selection from a matrix of potential synthetic targets is based on confidence in prediction of selected molecular properties prior to preparation and measurement. A comparable ability to predict relevant properties of organometallic compounds has, to date, not emerged from among the ranks of computational chemistry. This lecture will detail the development of a deeper understanding of the fundamental factors influencing key parameters of molecules for utilization in the CVD of electronic materials, and discuss the progress made thus far on the subject.

By its fundamental nature, chemical vapor deposition (CVD) generally is considered a thermodynamic process. In this manner, chemical bonds between atoms in precursor molecules are ruptured prior to or during, film growth, or perhaps, in both time regimes. Such bond scission typically is induced thermally, although alternate modes also have been employed. A principle tenant of CVD thus has been to presume complete cleavage of interatomic interactions in sources. In general, thermal operating conditions for most CVD reactions are sufficiently high to support this assumption. In recent years, however, needs have evolved which demand lower deposition temperatures. Key drivers for this emerging area include unique properties of metastable phases, thermally sensitive substrates, reduced CTE mismatch strain, and diminished thermal budgets associated with each step in device preparation. One consequence of growth at lesser temperatures is the possible introduction of undesired atoms in the deposited layer. Often these impurities arise from incomplete precursor decomposition. It has been observed, therefore, that some organometallic compounds provide a source for deleterious carbon incorporation. Presumably the origin of this observation is the complete thermolysis of metal-carbon bonds, either alone or in combination with secondary organic fragmentation from the original source molecule. The net consequence of deposition at such non-thermodynamic conditions is a breakdown of the operational paradigm involving complete bond rupture in precursors.

Several years ago, an alternative model of CVD was introduced. The basic premise of the "single-source" approach to precursors was the retention of relic molecular architecture in the film deposited from a precursor containing more than one element of the ultimate composition. In general, the bonds were present directly between the elements of interest. Such interactions tended to be the

thermodynamically weakest in the molecule. Operation at traditional thermal regimes, therefore, generally provided sufficient energy for cleavage of the bonds. Compensations for this have been to explore lower substrate growth temperatures, as well as to exploit the enhanced stability of leaving moieties from the core atoms, often by decorating the periphery of the central unit(s) with species possessing weak bonding interactions themselves. In reflection, however, few significant advantages have been discovered to date for CVD from unimolecular precursors, as opposed to the traditional employment of separate sources for each element in the final deposit. Nevertheless, there are instances when available evidence points toward the juxtaposition of atoms in the solid state which also were adjacent in the precursor.

In the case of doping of semiconducting materials, one need only incorporate relatively small quantities of the sought-after element into the host lattice in order to accomplish the desired effect. Thus, it was of interest to explore the possibility that unimolecular precursors might find utility as dopant sources. This was interesting, particularly in view of the dominant mode for current dopant introduction, ion implantation, which intrinsically is a thermodynamic process. Inherent in the unimolecular dopant approach is a shift toward the regime of kinetic growth.

This lecture will focus on the underlying principles of precursor molecular design, with an emphasis on the kinetic preparation of blue optoelectronic materials.

Electronic Structure Of UHV Grown C₆₀/Conjugated Oligomer Multilayers

**Carlo Taliani, Roberto Zamboni, Robert Marks,
Michele Muccini and Reiner Mahrt*.**

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** Phillips Universitaet, Fach Phys. Chem., Marburg*

A large attention has been devoted to the study of electron transfer in C₆₀/conjugated polymer composites but a detailed understanding of the basic processes is still debated. We will report on the UHV growth and characterization of multilayers of C₆₀ oligomer where the layers are defined at monolayer resolution with the aim to study the physics of the electron transfer process in a well-defined model system. The electronic structure is studied by means of absorption and photoluminescence spectroscopy. The dynamics of photoexcitations is studied by means of transient spectroscopy.

*Symposium [I]
13:45-15:45, Jan. 8*

Novel Polydiacetylene-Containing Polymeric Systems and Their Third Order Nonlinear Optical Properties

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In this lecture the results of recent studies on third order nonlinear optical (NLO) properties of two novel polydiacetylene(PDA)-containing systems will be presented.

First, the semicrystalline deep blue films of the irradiated poly(*m,m'*-butadiynylene sebacodianilide) abbreviated as PSA, were impregnated with *N*-methylpyrrolidone (NMP) and their third order nonlinear optical properties were investigated. PSA gives semi transparent films by casting, which developes deep blue coloring on irradiation. However, due to semicrystallinity, the films scatters light. It was found that impregnation of NMP improves considerably the optical quality of the films. The $\chi^{(3)}$ values of these films determined by the DFWM method at 532 nm were in the range of 10^{-10} esu. The films impregnated with NMP containing lanthanide metal salts showed $\chi^{(3)}$ values of the order of 10^{-9} esu. These values are somewhat enhanced by resonance as the films absorb 532 nm. Therefore, measurements using a non-absorbant wave length are being carried out and the results will be presented.

Secondly, composite systems consisting of nanocrystals of PDA and amorphous host polymers, will be discussed. For example, *N,N'*-di-*n*-butyloctadiynylene diurethane microcrystals were dispersed in a copolymer of vinylpyrrolidone and vinyl acetate, and cast films were irradiated with UV light, and extremely transparent deep red films were obtained. These films also have $\chi^{(3)}$ values of the order of 10^{-10} esu.

The relationships between the morphology of these films and NLO properties determined by THG using non-absorbant wave length will be discussed, as well as the effect of lanthanide metals salts on the $\chi^{(3)}$ values.

*Symposium [I]
13:45-15:45, Jan. 8*

Neutral and Doped States of Polymers with Aromatic Rings Studied By Resonance Spectroscopies

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Recent development of light emitting diodes has considerably increased the interest in conjugated polymers such as polyphenylene-vinylene (PPV) or polyparaphenylene. To fabricate reliable devices and improve their lifetime, the optimization of the properties of such compounds is required, in terms of conjugation length, defects control, ..to name a few. In addition, principal optical and electronic properties may be varied in a controllable way by an appropriate ring substitution. Also, a large number of studies have been devoted to oligomers, which can be considered as short chain models of conjugated polymers, and lead, in many cases, to better understanding of the phenomena occurring in high molecular weights compounds.

In this work, we have undertaken detailed spectroscopic investigations on both polymers containing aromatic rings, i.e PPV and PPP and their oligomers. Two main resonance techniques, Raman Resonance Scattering (RRS) and High Resolution Solid State ^{13}C NMR have been used with the goal to characterize the modifications in the electronic distribution of the chain backbone when a transition from the neutral to the doped state is observed. These two techniques are probes of the electronic system by providing information on the bond strength on one hand (RRS) and on the electron localization on the other hand (HR ^{13}C NMR).

In particular, we have studied the following compounds: neutral and sodium doped PPV, PPP and their oligomers. Using RRS, we have demonstrated the presence of quinoid-like segments of different lengths, showing in the same time that the identification of polaronic and bipolaronic segments is possible. In HR ^{13}C NMR, we have detected in PPV the carbon atoms which are the most affected by the carriers, as well as two types of Na species in polymers doped to saturation. These experiments clearly put in evidence the complementarity of these two resonance techniques to probe the nature of the chemical bonds of the polymeric chains before and after doping, via a modelization using a valence force field model.

*Symposium [I]
13:45-15:45, Jan. 8*

Solution Properties of Conducting Polymers

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Solutions of polyaniline and polypyrrole were prepared by dissolving the corresponding polymers in *m*-cresol and NMP. The solution properties of the polymers, such as inherent viscosity and hydrodynamic radius, in these solutions were measured by viscometry and light scattering techniques. Conditions required to make soluble polypyrrole will be discussed. Free standing film of these polymers were cast from the corresponding solutions. The electrical, thermal, and other physical properties of solution-cast polyaniline and polypyrrole films will be discussed in conjunction to these solution properties. Inherent viscosity and conductivity values of polypyrrole indicates that higher molecular weight polymer is obtained with higher oxidant/monomer ratio. However, the solubility of polypyrrole is not determined solely by its molecular weight. Conductivity data indicates that soluble polypyrrole consists more of polarons than insoluble polypyrrole, i.e. oxidation state plays an important role in dissolution of the polymer. Apparently, solubility of polypyrrole is determined by the molecular weight, oxidation state, and degree of crosslinking. ESR data shows that polypyrrole powder containing more of the soluble forms has a higher spin concentration than that of the insoluble polypyrrole.

UV-Vis spectra of polypyrrole solutions in *m*-cresol and in NMP were quite different from those of the corresponding solid. As the solvent being changed from NMP to *m*-cresol, the broad peak at ca. 440 nm (2.82 eV) increased at the expense of a sharp peak at 475 nm (2.62 eV). It is expected that, in an acidic solvent such as *m*-cresol, polypyrrole can be doped more favorably with an acidic dopant, including DBSA. On the other hand, in a basic solvent such as NMP, polypyrrole is presumably in the undoped neutral state. Therefore, the sharp peak at 475 nm and the broad peak at 440 nm correspond to $\pi \rightarrow \pi^*$ transitions in the neutral and doped forms, respectively. The conductivity data also support this argument. Polypyrrole film cast from NMP showed very low conductivity ($\sim 10^{-10}$ S/cm) while the film cast from *m*-cresol showed a conductivity of 1.3×10^{-4} S/cm. The peak absorption at 933 nm (1.33 eV) arises from the transition between defect states. For polyaniline, the effect of chain conformation on the electrical conductivity will be discussed.

Symposium [J]
16:00-18:00, Jan. 8

Polyurethanes for Second-Order Nonlinear Optics

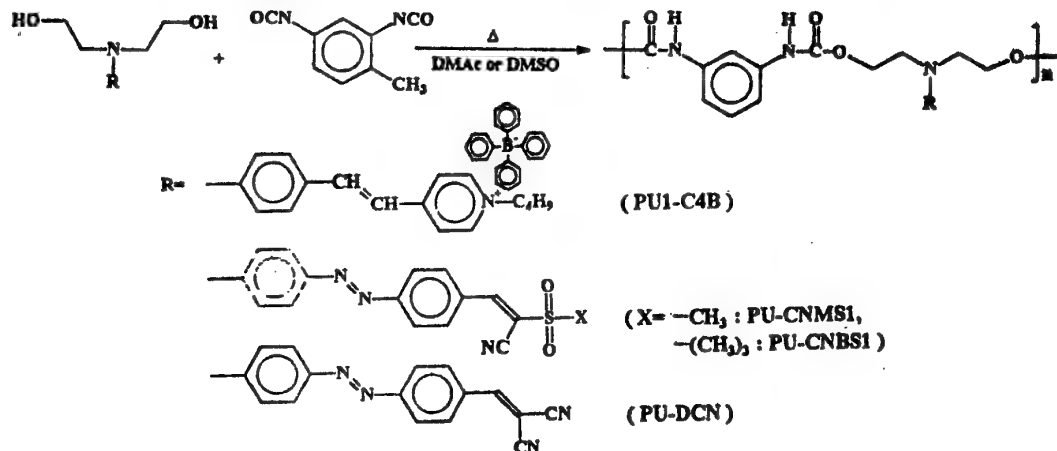
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Nonlinear optical (NLO) polymers have attracted much scientific and technological interest because of their potential application in the field of optical communications, optical processing, and optical computing. From the beginning stage of the NLO polymeric materials research in the middle of 1980's, thermal relaxation and low values of NLO coefficients of chromophores in these systems were the major obstacles for practical applications. Since then there has been a continuous efforts toward reducing thermal relaxation and increasing NLO activity of the materials. In recent years, in addition to enhancing the two fundamental factor, research was also extended to improve optical quality, processibility and chemical stability of NLO materials, which are the properties closely related to those of matrix polymers. Recently we have developed new polyurethanes with covalently linked side chain moieties containing a highly active NLO chromophores.



The main reason we selected the polyurethane as a matrix was that an extensive formation of hydrogen bonds between the urethane linkage is expected, thus increasing the rigidity of the matrix and to prevent the relaxation process of the oriented NLO chromophore dipoles. In this paper, we report the synthesis and characterization of polyurethane-based NLO systems. We also present the details of their optical second-order activity in terms of electric field poling, and temporal and thermal stability.

Symposium [J]
16:00-18:00, Jan. 8

Chirality Effects in Second-Order Nonlinear Optics of Thin Polymer Films

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Over the past few years the nonlinear optical properties of chiral materials have been extensively investigated. In particular, second-order nonlinear properties of chiral surfaces and thin films have been studied by several groups. Compared to the linear optical properties of chiral materials, the second-order properties of chiral surfaces have important differences.

Chiral surfaces show a different response to left- and right-hand circularity-polarized fundamental light for second-harmonic generation. In our work we have concentrated on understanding and separating the various physical mechanisms that can give rise to this nonlinear circular difference (CD) response from chiral materials.

In our experiments, we have utilized thin films of chiral poly(isocyanide)s functionalized with nonlinear-optical chromophores. The CD response of second-harmonic generation from chiral surfaces can occur within the electric-dipole approximation. However, it is not possible to explain satisfactorily our experimental results within the electric-dipole approximation. Chiral molecules are known to possess also significant magnetic dipole transitions. We have developed a technique to separate the electric and magnetic contributions to the observed surface nonlinearities. The technique relies on measuring the second-harmonic signals as functions of the rotation angle of a quarter wave-plate that controls the state of polarization of the fundamental beam. For the case of poly(isocyanide) thin films the largest components of the surface susceptibility tensor including magnetic transitions were of the order of 20% of the electric-dipole-only components.

The CD response of surface second-harmonic generation is analogous to the linear optical activity effects. However, we have shown that appropriately chosen linear input polarization can also be used to probe surface chirality. The measured difference effects between the appropriate linear polarizations are comparable to the CD effects. The fundamental origin of the linear-difference effects is the broken symmetry between the s- and p-polarized components of the fundamental light in the surface geometry.

The poly(isocyanide) films have in-plane isotropy. For such samples these nonlinear optical activity effects can occur only for chiral surfaces, i.e., for surfaces that have no mirror planes perpendicular to the surface. However, we have also used these techniques to investigate anisotropic achiral surfaces. As an anisotropic sample we have used Langmuir-Blodgett films of DCANP (2-docosylamino-5-nitropyridine) with the achiral C_{1h} symmetry. These samples were found to exhibit nonlinear optical activity (circular-difference and linear-difference response). However, in this case the optical activity arises from the chirality of the experimental setup. For most orientations of the sample, the experimental arrangement possesses no mirror planes, because the sample director, surface normal, and the wave vector of the fundamental beam are not coplanar, i.e., a coordinate system based on these vectors possesses a definite handedness. The optical activity was found to vanish for sample orientations that make the experimental geometry achiral. Furthermore, the optical activity effects were found to reverse sign as the handedness of the experiment was reversed.

Optimization of Molecular Architecture for Nonlinear Optics

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Conjugated polymers have attracted a great deal of attention in the domain of nonlinear optics due to the high hyperpolarizability of their π electron orbitals. Along the conjugated polymer chains the oriented polyacetylenes show the highest non resonant third order susceptibility and until recently no major improvement of the off-resonant susceptibilities on new molecular systems has been reported. However near resonances the nonlinear optical susceptibilities are highly enhanced. In addition, in conjugated polymers the resonant electronic excitations are strongly coupled with the backbone vibrations, i.e stretchings, torsions etc.. Therefore the study of nonlinear excitations with geometrical relaxations is of crucial importance not only to provide useful information for future devices with ultra-fast responses but also to elucidate fundamental aspects of the molecular dynamics in the solid state molecular systems.

In this paper we will discuss results of molecular dynamical studies in well defined oriented polymers as well as molecular engineering on rigid rod conjugated polymers. The use of ultra fast spectroscopy techniques to investigate instructed polymers give access to the basic relaxation mechanisms associated with elementary excitations. We used well oriented samples of polydiacetylene thin films to understand the relationship between structural characteristics at the molecular level such as the role of defects or conformational distortions and the delocalized electronic states as well as the molecular motion. We will give some examples of morphological modifications on oriented thin films that manifest optically. In addition the structure and morphology of the film surface at the nanometric and micrometric scales have been performed by AFM imaging. The study of the optical response to femtosecond optical pulses on the same samples show the time evolution of the vibronic spectrum of the conjugated backbone. On the polydiacetylene PDCH films made under controlled growth, the time resolved spectroscopy measurements give information on the quality of the polymer chains. The studies of vibronic coupling and the evolution in time of the different Raman modes give a better understanding of the electron delocalization dynamics in the conjugated chain which are not obtained by the static absorption spectra. For the samples grown on a hot crystalline substrate, a significant red shift as well as an increase of the oscillator strength of the excitonic $\pi \rightarrow \pi^*$ transition occur.

Stimulated by these investigations of the relaxation process in well known polydiacetylenes we started to focus our attention on the synthesis of new conjugated polymers with reduced molecular motion based on a rigid rod like character. We will report the synthesis and the characterization of various homo and copolymers based on the p-phenyleneethylene system. These compounds display high conjugation and appreciable $\chi^{(3)}$, exceeding 10^{-10} c.s.u.ⁱ. We will point out the effect of electron-donor and electron-acceptor groups on the enhancement of the nonlinear properties.

*Symposium [K]
10:30-12:20, Jan. 9*

Optical Computing With Bacteriorhodopsin Films

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The photochromic protein Bacteriorhodopsin (bR) is receiving much attention as an interesting alternative to conventional inorganic semiconductor based materials for a variety of applications in optical recording and information processing. Recent advances in genetic engineering techniques for tailoring molecular properties without degrading of its inherent mechanical and thermal stability make it an outstanding photonic material.

We exploit the parallel processing nature of optics and the real-time nature of bR films for implementation of several photonic applications. We demonstrate all-optical logic AND and Or gates. A two-color backward degenerate four wave mixing geometry with wild-type and chemically stabilized films of bacteriorhodopsin constitute the experimental setup. The saturation intensity, sensitivity and excited state lifetime (M state lifetime) of the films are very different. We use red light to form a holographic grating due to the M transition and blue light to form a grating due to the fast photochemical transition from M to B. Each of the two wavelengths in the experimental system acts as an input to the all-optical gates and the phase conjugate signal beam is the output of the gate. We developed an optical pattern recognition system using Optical Fourier Transform with bR films. The photo induced dichroism and its dependence on light intensity allows one to use the bR films as a real-time self-adaptive spatial filter at the FT plane. We demonstrate edge enhancement, and beam shaping.

*Symposium [K]
10:30-12:20, Jan. 9*

Dynamical Study of The Grating / Hologram Formed in Polymer-Dispersed Liquid Crystal Films

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We have developed an economic liquid crystal-polymer dispersion material that can be used for storing optical holographic images. In this paper, we demonstrate the use of thin samples of this material for such an application. Further we studied the dynamical behavior of the formed grating/hologram on the samples. The results show there are three mechanics involved during the formation of the grating / hologram. They are the degenerate four-wave mixing (DFWM) through the thermal effect, the spatially varying molecular weight of the different polymer molecules and the growth of the liquid crystal droplets. The details will be reported.

This work was supported by the National Sciences Council of the Republic of China under the grant No. NSC 85-211-M-006-011

*Symposium [K]
10:30-12:20, Jan. 9*

Preparation and Characterizations of Regularly Alternating Multiple-Quantum-Dots

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We have prepared and characterized soluble copolymers in which low band-gap sequences (thienylenic segments) regularly alternate with high-gap portions (phenylenic segments) linked by azomethine moieties therefore maintaining the overall π -conjugation. The polymer series with three, six and eight thienylenic residues showed interesting non linear optical properties together with evidences of electronic confinement in low-gap segments, superlattice structure, clearly envisaging applications as LED or photovoltaic devices. The products from polycondensation reaction of dialdehydes (in excess) and diamines end with carbonilic groups, hence they can be used in nanostructured substrate preparation (hydrophilic substrate), as already checked with starting dialdehydes self-assembled onto quartz slabs. These self-assembled structures can be obtained either by evaporation in high vacuum or by a proper thermal treatment, depending on the molecular weight of the molecules. As an example, it is noticeable that dialdehydes can be arranged in different inclination with respect to the substrate as a function of the film preparation procedure.

Self-assembled structures with higher molecular weight materials using sublimation techniques could be obtained using depositions in ultra-high vacuum chamber.

To evidence the superlattice nature, hence multiple-quantum-dot formation in the copolymers, Raman scattering measurements have been carried out, showing an electronic confinement in thienylenic blocks. Moreover, for these superlattice structures, aside from the main absorption peaks of two constituting segments in the UV-vis spectrum, other low-energy absorption have been predicted like in inorganic multiple-quantum-wells. These bands can not be evidenced, up to now in our copolymers, due to the intermolecular interactions and molecular weight distribution resulting in a broadening of the absorption peak.

As recently shown for a three-block conjugated compound, which constitutes a single quantum well structure, its dispersion in a proper matrix offers the way to observe the above cited features.

Blends of these copolymers in PVK and distributed PAT have been prepared. moreover matrix assisted polymerization of these polyazomerization using PVK and PVP have been carried out and sol-gel polymerization are also in progress.

Optical characterization of these materials will be presented and discussed in view of the above considerations.

*Symposium [L]
14:00-15:40, Jan. 9*

Novel Organometallic Compounds for Non-Linear Optics

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Interactions between π conjugated electrons and transition metal d electrons are receiving increasing attention due to the interest for the electronic and non-linear optical properties of metallorganic compounds. Indeed, the incorporation of metal atoms in conjugated molecules enhances the non linear optical response due to the presence of a low-lying charge transfer transition (CT).

Among organometallic compounds, oligothiophenes (T2 c T3) substituted with a Fisher carbene moiety ($\text{EtO-C}=\text{M}(\text{CO})_5$, $\text{M}=\text{W, Cr}$) is particularly interesting, as it combines the characteristic non-linear properties of the oligothiophene family with the effects of the transition metal substitution. In these molecules, a strong charge-transfer transition originating from the electron density transfer between the metal atom and the thiophenic backbone is observed at about 2.5 eV.

The electronic states of these intramolecular CT complexes have been investigated using semi-empirical quantum mechanical calculations which include the effect of the strong spin-orbit interaction associated with the d electrons. Optical linear and non-linear properties of these compounds have been studied using various spectroscopical techniques, both cw and time-resolved.

Radiative recombination is completely suppressed and resonant Raman spectra indicate that the CT electronic transition is strongly coupled with the metal-carbenic carbon stretching vibration. Transient spectra obtained by optical excitation at 3.2 eV show a strong bleaching of the CT band combined with an excited state absorption at about 2 eV. The reversal of the direction of the dipole moment upon excitation is responsible for the solvatochromism observed upon changing the solvent polarity. This effect leads to an estimate of the second order susceptibility using a simple two-state model.

*Symposium [L]
14:00-15:40, Jan. 9*

Ultrafast Relaxation Times by Time Resolved Degenerate Four Wave Mixing with Incoherent Light

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We present experimental results on the measurement of the population relaxation times of the excited states of some metalloporphyrins such as Zinc meso-tetra-phenyl benzporphyrin (Znm TBP), Zinc meso-tetra-phenylporphyrin (Znm TPP), Cobalt tetra.....porphyrin (CoTTP), Nickel tetra-tolly-porphyrin (NiTTP), Tetra-tolly-porphyrin (TTP) using time resolved degenerate four-wave mixing with the incoherent emission of a nanosecond pumped dye laser. The experiments are carried out in the counter propagating geometry ($K_4 = K_1 - K_2 + K_3$), where K_1 and K_2 are counterpropagating to one another and K_3 is at angle θ (90° with respect to K_1). Excited state and ground state relaxation times are calculated based on the theoretical formalism developed by Hiromi Okamoto (J. Opt. Soc. Am. B-10, 2353(1993)). The signals are recorded by delaying probe beam (K_3) for different fixed delays of the backward pump beam (K_2). We observe strong signal at a second peak due to the coherence of beams K_2 and K_3 for samples exhibiting excited state absorption and a narrow peak corresponding to the coherence of beams K_1 and K_3 for samples that do not exhibit excited state or reverse saturable absorption. The nonlinear absorption is studied using the Z-scan technique. From the experimental data we can estimate the relaxation times of excited states.

*Symposium [L]
14:00-15:40, Jan. 9*

Investigation of The Second and Third Order Nonlinear Optical Properties of Some Fused Heterocyclic Aromatic Compounds Related to 8-Hydroxyquinoline and The SHG Properties of Some of Their Metal Complexes

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We reported here the synthesis and the SHG properties of several metal complexes of 8-hydroxyquinoline and some of its derivatives. Few of these complexes gave modest second harmonic intensities in the range 0-30 times that of urea. The results of this study are compared with those obtained for other metal complexes and general trends are deduced. The compound bis(5-chloro-8-hydroxyquinoline) palladium(II) was shown to have the ability of second harmonic generation which is 30 times that of urea. In addition we also report the results of our coupled perturbed Hartree-Fock (CPHF) ab initio extended basis set calculations on the geometric structures and static first-order (α) and second-order (β), and third-order polarizabilities (γ) of a series of fused heterocyclic aromatic compounds. The effects of the introduction of a hetero atom in the ring system and other substituents (OH, NH₂, NO₂) on the first-order, the second-order and the third-order polarizabilities are described. Furthermore, the effect of the presence/absence of the heteroatom is presented.

*Symposium [L]
14:00-15:40, Jan. 9*

High Performance Materials

Designing Nanostructures Materials Success and Challenge

B. B. Rath

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Revolutionary changes in materials synthesis, processing and fabrication and characterization concepts has brought new challenges to systems design with improved performance and miniaturization. Recent advances have demonstrated that materials with nanometer scale structures can be successfully fabricated at the atomic and molecular levels by many methods to exhibit unique properties. Improved and unexpected properties of these materials impact a wide spectrum of phenomena including superconduction, magnetism, quantum electronics, nonlinear optics, cluster stability, and nucleation and growth. Concurrent with innovative processing and fabrication, new characterization tools such as scanning tunneling microscopy, atomic force microscopy and magnetic force microscopy have revolutionized our understanding of interatomic interactions and structures of atomic and molecular self organization. Selected observations of recent studies in this emerging field will be recent with recommendations for future research.

*Symposium [A]
13:20-15:40, Jan.5*

Recent Trends in Air Force Polymer Development

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As polymer technology has advanced over the last two decades, the opportunities for employment of advanced polymers in a wide variety of structural and non-structural applications have increased significantly. This has been particularly true for aerospace applications where high performance polymers are playing an increasingly significant role in primary structure and opto-electronic applications. Although there are a number of commercially available polymeric systems being successfully used to great advantage in both military and civilian aerospace systems, there is a continuing need to advance high performance polymer technology to meet critical Air Force needs. This presentation will detail some of the polymer research efforts carried out within the Polymer Branch and will reflect recent trends in Air Force polymer development. Earlier polymer research efforts directed toward advanced composite development will be discussed including the reinforcing fibers (poly-p-phenylenebenzobisoxazole and poly-p-phenylenebenzobisthiazole) and the matrix resin (benzocyclobutene oligomer). Research on aromatic-heterocyclic polymers for high temperature transparency development which resulted from previous fiber research will also be described as well as future directions currently being supported by the Air Force. These new areas include the most recent research efforts on opto-electronic polymers required for projected Air Force systems.

*Symposium [A]
13:20-15:40, Jan.5*

Future Opportunities and Direction in Advanced Materials

Andrew Crowson

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Materials research is leading to new classes of materials possessing increased strength and toughness, lighter weight, greater resistance to complex loading environments, and improved optical, magnetic and electrical properties. These advances are being achieved through the continual integration and understanding of the interrelationships between the microscopic and macroscopic domains in materials. The properties derived from such microstructures, covering a range of size and complexity, are in turn dependent on the synthesis and processing routes used to produce the materials. The Army Research Office (ARO) program in materials science is currently involved in the development of future generations of materials and materials processing technologies based upon this foundation. This is being accomplished by supporting research in six special thrust areas:

- (1) beam engineering/surface modification;
- (2) deformation/fracture phenomena;
- (3) strengthening and toughening;
- (4) synthesis and processing;
- (5) nondestructive characterization and
- (6) defect engineering.

This paper will describe these thrusts with reference to specific examples of future opportunities and directions foreseen in these areas.

*Symposium [A]
13:20-15:40, Jan. 5*

Photonic Materials for Space Applications

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Optoelectronic and photonic components offer a number of advantages, in terms of the speed, resolution, and the weight of the device, over their electronic components which make them potential candidates for application in space technology. However, the performance requirements of these devices in space environment are more demanding and stringent than that under normal ground based situation. One particularly important requirement is the radiation tolerance of the device. Ionizing radiation in the space environment alters the microscopic structure of the materials, thereby influencing the devices performance. This talk will discuss the materials requirements of photonic devices for space applications with an emphasis on the effect of ionizing radiation on the electronic and optoelectronic properties of materials.

*Symposium [A]
13:20-15:40, Jan.5*

Polymer-filled Glass and Star Gels: New Hybrid Materials

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A new process for generating polysilicates in nonaqueous media has enabled synthesis of polymer-filled glass composites with domain sizes on the nm scale. The underlying chemistry is the rapid reaction of formic acid with alkoxysilanes. A wide range of polymers can be accommodated. The polysilicate phase can become continuous at levels as low as 3 volume %, leading to substantial modification of system modulus and polymer viscoelastic behavior. These optically transparent materials can show high levels of toughness and yield behavior when the polymer is flexible. A second family of hybrid network materials -- the star gels -- results from single component molecular precursors. The molecules comprise an organic core with multiple flexible arms which terminate in network-forming trialkoxysilane groups. Clear, non-porous glasses which show brittle fracture but high levels of toughness have been generated from this family of precursors. Results from a variety of spectroscopic and mechanical investigations will be presented.

*Symposium [B]
16:00-17:50, Jan. 5*

The Properties of Poly(ethylene naphthalate) (PEN) and its Application

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TEIJIN Ltd. entered on the investigation of 2,6-naphthalene dicarboxylic acid (NDA) containing polymers in early 1960s. Since then, we have devoted a great deal of effort to the research and development of polymer design, monomer and polymer synthesis, and processing for them. Among them we finally commercialized Poly(ethylenenaphthalate) (PEN) in 1990 first in the world.

PEN shows excellent chemical and physical properties compared to conventional polyesters like Poly(ethyleneterephthalate) (PET) as follows;

- (1) Higher glass transition temperature (T_g)
- (2) Higher young's modulus
- (3) Improved hydrolytic stability
- (4) Enhanced gas barrier properties.

Now PEN proves to be highly prospective material as the base film for the Advanced Photo System (APS) and for magnetic recording media, as resin for returnable-refillable bottles and as fiber for paper machine clothing.

In this paper we describe the basic mechanism of property enhancement by introducing 2,6-NDA component into polyesters. The main features of our understanding for property- structure relationship for PEN are as follows;

1. Static stiffness: Higher aromaticity of naphthalene makes a chain stiffer with a large persistence length.
2. Dynamic rigidity: Bulkiness of the ring structure inhibits molecular motions. This dynamic rigidity, concerted with the static stiffness, plays a main role in the enhancement of glass transition temperature
3. Local structure: Large planar naphthyl moieties spontaneously form a local structure with parallel stacking of planes, which is similar to excimer structure.
4. High crystalline modulus: PEN has the planar zigzag configuration in the main crystalline form, which is expected to have a high theoretical modulus along chain direction.
5. High configurational entropy: Lower geometrical symmetry of naphthyl unit generates higher configurational entropy. This feature provides us with thermoplastic crystalline polymer with moderate melting point and high T_g.

We have used advanced molecular simulation techniques to elucidate molecular mechanism of the effect of introducing naphthyl unit, incorporated with extensive experimental works.

Synthetic Polymer Catalysts for the Hydrolysis of Phosphodiester

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Past three decades there have been great interests on the synthesis of enzyme-like polymers. A few synthetic polymers have been known to show catalytic activities, for example, imidazole-containing polymers being reported to catalyze the hydrolysis of phenylesters. While investigating polynucleotide analogues, we have found that the synthetic ribofuranose-containing polymers showed the catalytic activity for the hydrolysis of phosphodiester and the cleavage of oligodeoxyribonucleotides. This is the first example that synthetic polymers showed catalysis for the phosphodiester hydrolysis.

In order to elucidate the structural requirements for the catalytic activity, five polymers containing ribofuranose and pyranose rings were synthesized. Three of them showed the catalytic activity for the cleavage of DNA and the hydrolysis of phosphodiester with a rate acceleration of 10^3 compared with that of the uncatalyzed reaction. The catalytic activity seemed to be caused by the vic-cis-diol groups of furanose rings, which formed hydrogen bonds with two oxygen atoms of the phosphate so as to activate the phosphorus atom to be attacked by a nucleophile (H_2O).

*Symposium [B]
16:00-17:50, Jan.5*

High Performance Thermosets by Reactive Blending Technologies

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Most of the novel thermoset resins developed over the last decade are largely used as matrices in fibre-reinforced composites for the automotive and aerospace industries. As both military and commercial aircrafts are pushing towards greater speed, higher service temperatures are reached. Therefore an ever increasing temperature stability of these new resins is required which is obtained by increasing their rigidity and/or the crosslink density of their networks. However, in almost every case the same network which provides the high temperature properties also inhibits molecular flow, rendering the material low in toughness.

In the present contribution a number of different approaches are reported to enhance the toughness of tri- and tetrafunctional epoxies and of unsaturated polyester resins. In the case of epoxies we have employed as modifier, tough, thermally stable, engineering thermoplastics namely a bis-phenol A polycarbonate (PC) and a thermoplastic polyether imide (PEI). For the epoxy/PC blend a reaction was induced between the two components prior to the curing process while, in the second system the (PEI) component was simply dissolved in the uncured resin using a common solvent.

For unsaturated polyesters a different approach was adopted, a number of commercial liquid rubbers were chemically modified so as to enhance their reactivity towards the matrix functionalities. This in view of promoting a finer and more homogeneous dispersion and a better interfacial adhesion. A further system which will be described is aimed at mitigating another deficiency of the polyester matrix, namely its rapidly decreasing rigidity at temperatures above room temperature. In this case, a thermosetting Bismaleimide (BMI) was incorporated into the resin as a co-reactive second component. For all the investigated systems, molecular characterization was accomplished by FTIR spectroscopy. The fracture behavior was investigated at low and high rate of deformation using the linear elastic fracture mechanics approach. The results of this analysis were correlated eventually with the morphology of the fracture surfaces which was investigated by scanning electron microscopy.

Recent Development of Chemical Resistant Styrenic Polymers for The Refrigerator

Sang S. Woo and Kyung A. Ahm

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Environmental issue on ozone layer destruction is regulating the use of CFC11, which is known as a kind of Freon gas, depending on the economic level of each country. All the materials and processes using CFC11 have been destined for change. Refrigerator is an example. As a blowing agent, CFC11 has long been used. Recently HCFC-141B and cyclopentane are being used as alternatives. However, the former deteriorates the mechanical properties of current plastics and liners in refrigerator, while the latter does not harm the material but very explosive. Novel material is under development, but still not commercial. Feasible way is to increase the chemical resistance of the plastic parts, which is mostly made of ABS resin. In this talk, the change of blowing agents according to the environmental issue and new technology and trends to enhance the chemical resistance of styrenic polymers will be presented.

*Symposium [C]
10:30-12:30, Jan.6*

PBI Powder Processing to Performance Parts

O.R. Hughes

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A new route, direct forming, was developed for forming dense molded shapes from poly[2,2' phenylene)-5,5'-bibenzimidazole], PBITM, powder. PBI in molded forms exhibits exceptional thermal and mechanical properties.

Direct forming is a more productive alternative to hot compression molding. The new process affords the possibility of automated PBI powder shaping by cold compaction and densification in batches of multiple parts by powder-assisted hot isostatic pressing.

Two developments enable PBI direct forming: 1) the discovery that PBI powders that are porous and plasticized with moisture can be shaped by compaction at ambient temperatures (cold compacted), and 2) a finding that these cold compacted shapes can be further densified in large batches by a powder-assisted hot isostatic pressing.

Porous PBI powder is formed from PBI in solution by a spray-precipitation process. When plasticized with moisture this powder is cold compactible to PBI shapes with densities up to 94% of that of ultimate density of PBI. These shapes, which have sufficient strength to be handled are then further consolidated via powder-assisted hot isostatic pressing to shapes with excellent thermal and mechanical properties and densities of about 99% of the ultimate.

PBITM is a trade mark of Hoechst Celanese Corporation. PBI in molded shape forms is marketed under the trade name CelazoleTM.

*Symposium [C]
10:30-12:30, Jan.6*

A Correlation Study for The Mechanical and Thermal Properties of Gamma-Irradiated Low Density Polyethylene

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Mechanical testing and thermal gravimetric TG analysis have been applied to studying the effect of gamma-irradiation on the physicomechanical properties of low density polyethylene LDPE of the grade CDO230. The crystallinity is found to vary with the irradiation dose as well as with the elastic modulus, ductility and ultimate strength. The changes in the mechanical properties correlate with the change induced by the accumulation of stable radiation effect. The correlation between the tensile properties and TG data is limited to the tensile strength and softening temperature. The results are discussed in terms of perturbation, degradation and crosslinking of the polymer.

*Symposium [C]
10:30-12:30, Jan. 6*

Spectroscopic and Thin Layer Chromatographic Studies on Some Homo And Copolymers of N-Acryloyl Para Amino Benzoate

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The homo and copolymerization of N-para amino benzoate(NAPAB) with methyl methacrylate (MMA), ethyl acrylate (EA) and styrene(St.) were carried out by solution polymerization technique in the presence of azobisisobutyronitrile(ABIN) as initiator in sealed tube containing benzene as a reaction medium under nitrogen atmosphere at 60°C. The pattern of the chemical reaction is discussed. The monomer reactivity ratios were determined. The obtained samples were found to be alternating copolymer of azeotrope type. The UV-VIS spectra of homo and copolymer samples show three maximum absorption bands in the UV- region at 215, 240 and 270nm for M-M* transition (E_2 -band) in benzene ring and five absorption bands in the visible region at 350, 367, 655, 660 and 665nm, for n-M* transition of substituted benzene ring and due to the polymer color respectively. Infrared spectroscopic studies show some changes in the band position and intensity according to the polymer structure and assure the presence of methyl, methylene, methine, carbonyl, phenyl ring and NH groups at 2990, 702, 2910, 1718, 1599 and 3443 cm^{-1} respectively. Thin layer chromatographic separation of NAPAB-MMA copolymer samples which have the same specific viscosity, but differ in NAPAB content is achieved in normal saturation system using silica gel (G60) as stationary phase and butylacetate or dimethyl sulphoxide as mobil phase at 25°C. Also, thin layer chromatographic separation of NAPAB-MMA copolymer samples according to their specific viscosities is obtained by using a binary elution mixture of dimethyl sulphoxide as good solvent for the copolymer samples and methanol as non solvent (1:3 v/v) in normal saturation system at 25°C on the silica gel plates of 0.5 mm thickness. The mechanism of separation in both cases is discussed.

*Symposium [C]
10:30-12:30, Jan.6*

New Optical Polymers with High Stability of Nonlinear Effects

**Martin Doebler, Christoph Weder, Peter Neuenschwander, Ulrich W. Suter
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Department of Materials, Institute of Polymers.

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Aiming to prepare polymeric materials for electro-optical as well as photorefractive applications, a series of copolymers has been synthesized from an NLO-phore monomer, i.e., 2',5'-diamino-4-(dimethylamino)-4'-nitrostilbene, and a charge-generating monomer, i.e., 4-(bis-(2-hydroxyethyl)amino)-benzaldehyde-1,1-diphenyl-hydrazone. Three different types of systems were prepared: The monomeric units were enchaind randomly (random copolymers), in blocks (block copolymers), and as a blend of the two homopolymers. All films were amorphous and could easily be spun into films of optical quality and oriented by corona poling. Compared to their glass transition temperatures, these novel materials show the highest NLO relaxation times measured to date in polymers. Also, they exhibit photoconductivity in striking dependence of the morphology. Finally, some of these polymers are photorefractive.

*Symposium [D]
14:00-15:30, Jan.6*

Study of Energy Gap and Electronic Transitions in Model Polymer Systems: Electronic Versus Optical Excitations

**J.J. Pireaux, G. Gensterblum, C. Gregoire, B. Y. Han, K. Hevesi,
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The use of monochromatized electron beam to probe the electronic structure around the energy gap is not a widely spread research tool to study polymeric materials. Though, providing surface charging is well controlled, electron energy loss spectroscopy (EELS) could produce unbeatable pictures of the energy gap, the gap states/defects and the electronic excitations from the surface/interface of polymers. The technique could thus reveal itself very complementary to optical spectroscopies in probing surface-like versus bulk-like properties, although with different selection rules.

The potentialities for the study of e.g. metal-polymer contacts will be illustrated with three test cases:

- The evidence of irradiation induced defects in the energy gap of polyethylene, and in the secondary electron emission of hexatriacontane, a negative affinity material;
- The study of singlet and triplet excitons in single crystal C₆₀
- The electronic excitations in catenane thin films.

*Symposium [D]
14:00-15:30, Jan.6*

Non-Destructive Evaluation of Polymeric Bulk Materials and Coatings Using Two-Photon Confocal Microscopy

**P.C. Cheng, S.J. Pan, A. Shih, W.S. Liou,
J.D. Bhawalkar*, P.N. Prasad* and B.A. Reinhardt****

Advanced Microscopy and Imaging Laboratory,

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Confocal microscopy has been a commonly used tool to obtain three-dimensional (3D) images of biological specimens such as tissues and cells. In recent times, two-photon laser scanning microscopy using infrared laser light, has been attracting significant attention. Due to the larger penetration of infrared light in doped polymeric media and reduced photo-induced damage at longer wavelengths, this relatively new technique is a promising alternative for imaging polymeric materials. In addition, the quadratic dependence of two-photon induced fluorescence on excitation intensity results in excellent depth resolution. This talk will discuss the use of two-photon laser scanning microscopy as a nondestructive evaluation technique for polymeric bulk materials and coatings. We demonstrated that one can easily investigate the surface, the bulk, and any underlying fractures or defects by this technique. Images showing subsurface microfractures in the volume of a doped-polymer block have been obtained. By using different fluorophores in different layers of coatings, we have used multichannel two-photon laser scanning microscopy to selectively probe each layer. A major advantage of using IR excitation is that UV excitation normally used in single photon fluorescence microscopy could lead to chemical damage of the polymer coatings, whereas the IR excitation is relatively safe. Imaging was achieved in both positive and negative contrast modes. The fluorophores used were newly developed, highly efficient two-photon induced upconverters. This resulted in excellent signal strength at low pumping power.

The talk will be followed by a brief video presentation illustrating 3-D images obtained by this technique.

*Symposium [D]
14:00-15:30, Jan.6*

Improved Elastomers Through Control of Network Chain-Length Distributions

J. E. Mark

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The goal of primary interest in these investigations was the development of a novel method for preparing elastomeric networks having unusually good ultimate properties. The technique involves end linking mixtures of very short and relatively long functionally-terminated chains to give bimodal networks. Such (unfilled) elastomers show very large increases in reduced stress or modulus at high elongations because of the very limited extensibility of the short chains present in the networks. This non-Gaussian behavior also appears in compression or biaxial extension, shear, tearing, and cyclic fatigue tests. Non-Gaussian theories taking into account this limited extensibility give a good account of the experimental results. Bimodal distributions also facilitate strain-induced crystallization, and thus the reinforcement it provides. It is pointed out that some types of elastomeric networks described in the literature may have been bimodal, albeit inadvertently.

*Symposium [E]
16:00-18:00, Jan. 6*

Silicon-based Polymers as High Performance Materials

Itaru Mita

Dow Corning Asia, Research Center

Silicones, the most important silicon-based polymers, have been used as thermally stable elastomers and fluids for many years because of their excellent chemical thermal stability. Recently, many efforts are being made to obtain silicon-base polymers having, in addition to chemical thermal stability, higher physical thermal stability which may be used as high temperature materials or other high performance materials.

One direction of these efforts is the study on polycarbosilanes the main chain of which is less flexible than polysiloxane chain. Another effort is the use of aromatic substituents to make the polymers more thermally stable in both physical and chemical senses. Another direction is the introduction of the network structure or other non-linear structure to polysiloxane or polycarbosilane based polymers. Japanese government is funding a project on such silicon-based high performance materials. The development in this field will be reviewed. The fourth direction is the hybridization of silicon-based polymers with organic polymers which will be the topic of one session of this meeting.

*Symposium [E]
16:00-18:00, Jan.6*

Silicone Surface Science Opportunities

Michael J. Owen

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In some circles it has become fashionable to argue that the great days of scientific discovery and technological implementation are over. There is no room for this technophobic view in the expanding field of synthetic silicon-based materials. Business based on silicon science and technology is growing globally and particularly marked in applications dependent on surface properties and behavior of silicones.

Much of this established business and many future commercial opportunities depends on polydimethylsiloxane (PDMS), still the most important material industrially in the organosilicon-based materials field. Applications such as adhesion, release, foam stabilization, antifoaming, antifouling, de-emulsification, surface protection, etc all depend in one way or another on the properties of PDMS interfaces. The attributes of this polymer that make it such a valuable surface active material will be reviewed with particular emphasis on the four most fundamental characteristics: low intermolecular forces between methyl groups, high chain flexibility of the siloxane backbone, high siloxane bond energy, and the partially ionic nature of the backbone. The first two explain much of the physical behavior of PDMS whereas the other two account for the chemical consequences of the PDMS structure.

From these considerations PDMS emerges as a particularly favored case of a very low intermolecular force pendent group arranged along the most flexible backbone available, thus enabling the methyl groups to be presented to their best effect. These factors combined with the bonus of considerable thermal and oxidative stability account for the multitude of PDMS surface applications. A broad overview of new developments and global trends in surface-active, low-surface-tension materials, emphasizing PDMS will be presented to illustrate this diversity.

*Symposium [E]
16:00-18:00, Jan.6*

MESO Modelling in Liquid Crystalline Polymers

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Thermotropic liquid crystalline polymers have great potential as structural materials. They have high strength and stiffness in the direction of molecular alignment and their low melt viscosity facilitates processing. Without resort to large aligning fields, moulded samples show inhomogeneities either in the form of variations in the direction of alignment across the sample or topological defects. An understanding of these textures and defects is crucial to realizing the full potential of the polymers.

The microstructures observed in LCPs are different from those observed in small-molecule nematics because of the anisotropy of the Frank elastic constants in the polymeric case. In polymeric nematics, the splay constant is the highest and the twist constant the lowest. Splay distortion requires chain ends to segregate to maintain a constant density and this organization is entropically unfavorable.

A modelling technique has been employed to investigate the effect of different elastic constants on microstructural form. Previous models and simulations have worked primarily in the single constant approximation which is valid in small molecule nematics but not for polymeric liquid crystals. The new technique separates the contributions from splay, twist and bend by discretising Frank's equation onto a 3D array of cubic cells. A Monte Carlo algorithm is used to find minimum energy configurations with respect to defined boundary conditions and to study the evolution of microstructure in the bulk.

Modelling has shown that the character of the microstructures which occur during the evolution varies a great deal with the values of the Frank elastic constants put into the simulations. When elastic constants appropriate for polymeric liquid crystals are used, simulated microstructures show good agreement with experimentally observed microstructures.

While it is particularly challenging to extract three dimensional information from experimental observations, the 3D simulations are readily interrogated to observe the nature of defects in the bulk. Algorithms have been written to find topological defects within the simulated volumes and to characterize them in a quantitative fashion.

*Symposium [F]
10:30-12:30, Jan. 7*

Synthesis of Block Graft Copolymers Containing Liquid Crystalline Segments

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Block and graft copolymer having LC and amorphous or semicrystalline segregated domains have received great attention due to their potential for some new technological applications, as well as due to their uncommon morphology originated from a combination of the two main ordered state of matter, the mesomorphic short range order of the LC moieties and the long range compositional order at a supramolecular level of the phase segregated block and graft copolymer system. Several attempts were done to produce these materials and they consist mainly in living ionic or living insertion polymerizations by step addition of common and LC functionalized monomers, or in chemical modification of starting block copolymers by Low Molar Mass Liquid Crystals with reactive end groups. Our synthesis goals to the synthesis of block and graft copolymers are based on:

- (i) Radical polymerization of a liquid crystalline acrylate (LC monomer) onto a modified EVA matrix functionalized with thiol groups as macrotransfer agents;
- (ii) Chemical modifications of poly-(styrene-*b*-acrylic acid), ionomers and *t*-butyl ester derived therefore by low molecular weight liquid crystals with hydroxyl, bromine and tosyl end groups;
- (iii) Radical block copolymerization between styrene and a LC monomer by ININFERTER techniques.

The block and graft copolymers produced were thermally and structurally characterized, and comparisons were made in relation to the incorporation degree of LC chains onto the precursor matrices.

*Symposium [F]
10:30-12:30, Jan. 7*

Using Computational Chemistry To Aid The Design of Contact Lenses

B E. Eichinger and David Rigby

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Hydrogel contact lenses have been an important optometric product since the discovery of the hydrophilic monomer hydroxyethyl methacrylate (HEMA). Continuing improvements in lens materials beyond HEMA are being sought to further enhance comfort, extend wear, and discourage spoilation. The contact lens industry has undertaken significant research programs to achieve these goals, but the rate of introduction of new products on the market has been disappointing. It is difficult to find the delicate balance in a polymeric material that swells in tear fluid to the desired extent, has good O₂ permeability, good mechanical strength, and which does not adsorb the lipids, proteins, or glycoproteins that are found in tear fluid.

Contact lenses are particularly simple examples of biomaterials, but they are subject to constraints imposed by the lens material interaction with a biological fluid. In this materials design problem the number of combinations to be tested in formulations can become very large indeed; there are well over 100 different monomers that can be tested, and we know of companies that are interested in testing copolymers having as many as six different monomers. The use of computational methods can help to guide this research by illuminating trends of behavior, predicting properties before materials are made, and by providing a disciplined scientific approach to the research effort. In application to copolymeric hydrogel contact lens design, computational methods provide estimates of:

- Relative stabilities of the radicals and/or ions that are generated in polymerization reactions and relative activation energies for estimation of polymerization rates.
- Relative solubilities of water, O₂, and other gases in polymers and in copolymers from estimates of heats of mixing.
- Rates of diffusion of H₂O, O₂, and other gases through polymer and copolymer solutions at any desired composition.
- Modulus of elasticity based on percentage of multifunctional monomers in the polymerization mixture.
- Molecular organization of polymer surfaces in the presence of surface treatment agents and tear fluid, and the mode of interaction of tear fluid components with polymer surfaces.

Each of these topics will be briefly discussed.

*Symposium [F]
10:30-12:30, Jan. 7*

Interpretation of Stress-Strain Isotherms Of Cis 1,4-Polyisoprene and Cis 1,4-Polybutadiene Networks Crosslinked at Different Degrees of Dilution: Comparisons With Theory

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Stress-strain isotherms for unfilled crystallizable polymer networks show upturns at higher elongations due to the strain-induced crystallization. These upturns represent a toughening of the elastomer, frequently placing it in the category of high-performance materials. The networks crosslinked in solution attain "simpler topologies", which consist of less degree of entanglements among the chains. Also, it is believed that the removal of the solvent after crosslinking collapses the chains into a type of compressed or "super-contracted" state. In the present study, the constrained-junction theory is applied to the strain-induced crystallization of polymer networks crosslinked in solution with different dilutions. A recent investigation (Sharaf, M. A.; Kloczkowski, A.; Mark, J. E.; Erman, B. *Computational Polymer Science*, 1992, 2, 84) showed that the rescaling of the molecular deformation from the macroscopic to molecular level in constrained-junction theory gave stress-strain isotherms that were in satisfactory agreement with experiment. They represented improvements in the predictions of the theory of the strain-induced crystallization developed by Flory. The degree of crystallinity and the melting point (which increase with deformation) and the reduced stress in solution-crosslinked networks were calculated using this model. The severity of chain entanglements, as measured by the parameter κ , approached the affine limit. Generally, higher values of ζ indicates an increase in the nonaffine nature of the transformation of the domains of constraints with applied deformation. Values of the experimental small-strain modulus ($2C_1 + 2C_2$) at $\alpha \sim 1$ and those of the calculated affine modulus (νRT) were found to be in a very good agreement. Experimental values of the elongation α_{cryst} , where the upturn of the modulus begins, were in satisfactory agreement with theory.

*Symposium [F]
10:30-12:30, Jan. 7*

Design of Thermosets With Greatly Improved Properties.

J. Economy

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Recently we have reported a new family of thermosetting aromatic copolymers which display outstanding high temperature properties. As compared to epoxies or phenolic resins this new system has been shown to be at least 150°C more stable in air or nitrogen. Furthermore, the spent polymer can be recycled back to an oligomer which can then be repolymerized. In this talk, particular emphasis is placed on the fundamental and technological problems we have solved in tailoring this system for use as a high performance adhesive and low dielectric constant substrate for design of multi-ship module laminates.

*Symposium [G]
16:00-18:00, Jan. 7*

Solution viscosity of Network-Forming Polyurethanes

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Network-forming polyurethanes were prepared by the reactions of hexamethylene diisocyanate (HDI) with an oxypropylated triol (LHT 240) in bulk and in solvent (nitrobenzene). The resultant structures were rather compact compared with linear polyurethanes. The gelation studies revealed the established patterns of increased intramolecular branching with increased dilution of reactants.

Intramolecular reaction in such reactant systems produce ring structures of various sizes. The possible effect of these ring structures on the overall molecular dimensions were investigated from dilute solution viscosity measurements.

Ring structures were abundant in end-linking reactions of polyols and diisocyanates, although they were rheologically insignificant. The ring sizes were negligible compared with the molecular sizes of the network chains. Solution viscosity measurements complimented the traditional rheological characterisation of shear and normal stresses, and showed the universal relationship between intrinsic viscosity and molar masses.

*Symposium [G]
16:00-18:00, Jan.7*

Luminescence Studies of Polystyrene Derivatives Containing Grafted Chromophores

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Luminescent polystyrene derivatives in which the chromophoric groups were attached as pendant groups were synthesized. Examples are poly(stilbene-*p*-methoxy styrene), poly(styrene-co-stilbenyl-*p*-methoxy styrene) and poly(styrene-co-anthracenyl-3-methoxystyrene). In the stilbenyl based copolymers the monomer ratios between styrene and chromophoric units were 2:1 and 1:1, and in the anthracenyl based polymer the ratio was 2:1. A model compound, *p*-(*p*-ethylphenoxy)methyl stilbene was also prepared to provide information on the chromophore behavior in the absence of a polymeric backbone. Based on absorption and emission studies of the polymers in solution and in the solid state, and taking into account the model compound behavior a mechanistic scheme has been proposed in which chromophore aggregation in the ground state and energy migration both play an important role.

*Symposium [G]
16:00-18:00, Jan. 7*

Simulations on Thermoplastic Elastomers from Polypropylene

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Research has been done to investigate sequence lengths, and sequence-length distributions of stereoregular polymers. Computer simulations were also used to investigate the development of reinforcing crystallites in thermoplastic elastomers. This is done by constantly monitoring the sequence lengths, and sequence-length distributions. New types of stereoblock polypropylene that act as thermoplastic elastomers through controlled crystallization would be of particular interest in this regard. While it is widely believed that sequence distribution in these polymers is the primary cause, no theory was able to predict the characteristics of the thermal transition of polymers. Following up the Windle approach in generating copolymeric chains using Monte Carlo methods, one hundred chains have been simulated in order to allow for a search of crystallinity in these polymers. According to the amount of crystallinity found in these polymers at various feed compositions, the melting points of the different samples have been predicted. Other physical properties such as the interfacial free energy, the standard free energy of fusion and Young's modulus at small extensions are also predicted. The work is capable also for predicting the size of the crystals and the minimum sequence length required for crystallization.

*Symposium [G]
16:00-18:00, Jan. 7*

High Performance Protective Coating for Atmospherically Exposed Reinforced Concrete

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The deterioration of reinforced concrete in the hot, humid, salt-laden conditions has been well documented. This deterioration is caused by corrosion of steel reinforcement which results in cracking and spalling of the concrete and ultimately will result in failure of the structure.

Corrosion of the steel reinforcement is most commonly caused either by carbonation of the concrete and/or the ingress of chloride ions into the concrete.

The use of protective coating has been identified as an effective method of minimizing carbonation and reducing the ingress of chloride ions into reinforced concrete and is now widely practiced.

This paper represents the results on investigations into the performance of an Aliphatic Acrylic coating with a Silane-Siloxane primer which have been used on several concrete structures in the Middle East.

Based on the results of these investigations and other work, standard specifications for concrete protective coatings have been developed and are discussed.

*Symposium [G]
16:00-18:00, Jan. 7*

Template Polymerization of Dimethylaminoethylmethacrylate in The Presence of Poly (Acrylamide -co-Acrylic Acid) by Gamma Radiation and Its Use for Removal of Metal Ions

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The polymeric material of poly (acrylamide - acrylic acid - dimethylaminoethylmethacrylate) "PAM - AA - DMAEM" was prepared by gamma radiation induced polymerization of dimethylaminoethylmethacrylate (DMAEM) in the presence of prepared poly (acrylamide - co - acrylic acid) as a template polymer "PAM-AA" using a template polymerization technique.

The effect of gamma radiation on the polymerization process showed that the conversion increases with the radiation dose. The obtained polymeric complex material is a water-soluble polymer at low doses < 10 kGy. On increasing the radiation dose the polymer was converted to a gel with the swelling degree varying from 15 to 36. It was found that the capacities of the obtained polymeric gel towards cations of Cu^{2+} increases with increasing the absorbed dose, monomer concentration and the template polymer / monomer molar ratios.

Spectroscopic studies showed that the mechanism of the floc formation is due to the interaction between the polymer and the copper sulfate in a bond formation between the active group of the polymer chains and the ions of copper sulfate. The amide and tertiary amino groups form complex with Cu^{2+} while the carboxylic group interacts with the ion through an ion exchange mechanism.

*Symposium [K]
10:30-12:30, Jan.9*

FTIR and UV Spectra of Iodinated Poly(acrylic acid)- Poly(vinyl alcohol) Complex

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The interaction between poly(acrylic acid) and poly(vinyl alcohol) in aqueous solutions of different pH values was examined by using FTIR and UV spectroscopy. The analysis of the spectra showed that at low pH values (~ 3.2) an interpolymer complex is formed between the two polymers and the hydrogen bonding is the mechanism of interaction between them. At higher pH values (~ 11), blends of the two polymers are formed. The spectroscopic techniques were also implied for the investigation of the coordination phase of metal ions in a ternary metal-polymer complex of PAA-metal-PVA. The results revealed that the interactions between the metal and the polymers lead to the formation of ternary polymer-metal-polymer complexes. The effect of iodine on the structural properties of the blends and complexes were studied by following the induced changes in their spectral features.

*Symposium [K]
10:30-12:30, Jan.9*

Characterization of Heat-Treated Electropolymerized Polyacrylonitrile

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PAN samples have been prepared by collection from electrolytic solution at different current densities. The time of collection was 4 hours at room temperature. The samples were heat treated at temperatures between 100 - 600°C for two hours. Both IR and DC conductivity showed that conjugation of PAN depends on the current density and pyrolysis temperature up to 450°C. This is attributed to the structural changes in the matrix.

*Symposium [K]
10:30-12:30, Jan.9*

Artificial Neural Networks: A Novel Approach for The Analysis and Predictions of Mechanical Properties of 6063 Aluminum alloy

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6063 aluminum extrusion is produced in the Sultanate of Oman for local use as well as for marketing abroad and is extensively used for the preparation of windows as well as door frames for the houses and commercial centers. The age hardening treatment was applied to 6063 aluminum alloy with the objective to establish the pertinent variables of temperature and time duration resulting in distinct improvement of mechanical properties of the alloy. A novel approach using an artificial neural network model was developed to predict the optimum tensile strength, yield strength, hardness and % age elongation and to analyze these properties. The model was based on the data derived for these properties by varying time and temperature in different experiments. Results have revealed that aging between 8-10 hours at 448 K is the most suitable combination of time and temperature, imparting maximum tensile strength, yield strength and hardness to this alloy. Ductility, on the other hand has shown a reverse trend.

*Symposium [K]
10:30-12:30, Jan.9*

Electroless and Electrodeposition of Composite Coatings

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Metal Matrix composite coatings incorporating a variety of solid particles such as carbides, oxides and polymers are technically deposited from usual electroless and electroplating baths. The resulted coatings offer a number of unique useful properties regarding their physical, chemical and mechanical properties such as high hardness for abrasion resistance, low-friction for sliding parts, improved corrosion resistance, etc. The properties of composite coatings are governed by particle size, concentration, and mode of distribution. The mechanism of co-deposition of various particles SiC, Al₂O₃, quartz, sand, PTFE with Ni was studied in view of the electrokinetic charge characterizing solid particles. Means to improve the mobility of the particles in the plating solution were investigated using a surface-active agent. Factors affecting the deposition process have been investigated and reviewed. The surface morphology, hardness and wear resistance of the composite coating were determined. Sound, coherent, high wear resistance coatings could be produced.

*Symposium [K]
10:30-12:30, Jan.9*

Acid Corrosion Inhibition of Nickel By Some Organic / Macrocyclic Compounds

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The corrosion inhibition of Nickel in 0.5 M sulfuric acid solution by 2-(Tryphenyl phosphoranylidene) succinic anhydride (2TPSA), 1,4,8,11 tetraazocyclo-tetradecane (cyclam), and N,N'-bis (2-amino ethyl)-1,3-propane diamine (2,3,2 tet.) have been investigated using DC polarization technique.

The anodic polarization curves of Nickel in 0.5 M H₂SO₄ at different concentrations of the three inhibitors were recorded at room temperature. The passivation current density values were used to calculate the percentage inhibition for each inhibitor concentration. The results were compared with fits obtained from the application of Langmuir, Frumkin, Flory-Huggins isotherm, and also from the application of kinetic-thermodynamic model. The results obtained were correlated to the chemical structure and the macrocyclic effect of the inhibitors.

*Symposium [K]
10:30-12:30, Jan.9*

Synthesis and Properties of Poly(pyromellitimide)s and Poly(naphthaleneimide)s with (n-Alkyloxy)methyl Side Branch: A Comparison of 5- and 6-Membered Imide Rings

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Bearing in mind the presumption that a 6-membered imide ring could have higher thermal stability than a 5-membered one, a series of poly(pyromellitimide)s (PYPIs) and poly(naphthaleneimide)s (NAPIs) having various (n-alkyloxy)methyl side branch have been prepared by condensation of imidazole-blocked 2,5-di[(n-alkyloxy)methyl]-1,4-benzenedisocyanates with pyromellitic dianhydride and 1,4,5,8-naphthalenetetracarboxylic dianhydride, respectively. Their chemical structures were characterized by spectroscopic means and various physical and thermal properties were measured and discussed with respect to the structural difference between 5- and 6-membered imide rings. Contrary to our expectation the NAPIs were found to be less thermally stable than PYPIs as studied by TGA. Other properties like phase behavior, crystallinity and solubility reveal that the 6-membered NAPIs have generally more flexible main chain.

To investigate the origin of these phenomena synthesized N,N'-diphenyl and -dialkyl pyromellitimide, N-phenylphthalimide, N,N'-diphenyl, and -dialkyl naphthalenediimide and N-phenylnaphthalenemonoimide as model compounds. Compared to N,N'-dialkyl pyromellitimide both N,N'-diphenyl pyromellitimide and PYPIs showed a bathochromic shift in π - π^* absorption maxima, but such shift was not observed in 6-membered model compounds and polymers. Contrary to the chemical equivalency of both C=O groups of a soluble PYPI known in a literature ^{13}C -NMR spectra of the NAPI model compounds showed a splitting of C=O peak, indicating that two different structures must exist.

To link these experimental results with proper chain structure of NAPIs we conducted ab initio calculations on both the monophenyl model compounds. This calculation indicated that the three bonds tied with N atom have planar structure in 5-membered ring, while in 6-membered imide ring those have a trigonal pyramid structure.

The Effect of Sulfate Ions on the Optical and Electrical Properties of Low Density Polyethylene

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The optical and electrical spectroscopies of pure dry and immersed low density polyethylene (LDPE) were investigated. The optical absorption spectra were measured in the wavelength range from 200 to 1100nm while the a.c. conductivity was studied in frequency range $10^2 - 10^5$ Hz. These measurements have been carried out at various times of immersion for LDPE film in 10% solution of CuSO_4 .

The calculated values of energy gaps (E_{opt} and ΔE) were found to be time dependent. The data of a.c. conductivity were analyzed to obtain the d.c. conductivity (σ_{dc}) and the optical dielectric constant(ϵ_{∞}). The results showed that:

- (1) direct and indirect optical transitions are exist,
- (2) direct and indirect energy gaps were increased as time increased.
- (3) the Urbach energy band tail has large values and decreases with time,
- (4) the d.c. conductivity decreased as time increased. while the dielectric constant increased.

These results could be explained as the formation of voids and the degradation of the LDPE.

*Symposium [L]
14:00-15:30, Jan.9*

A Study on Corrosion Resistance Characteristics of 6063 Aluminium Alloy

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The aluminium alloys in marine application includes structures, pipelines, and boats etc. and shows good resistance to seawater. The aluminium-magnesium-silicon alloys are some what less resistance to sea water. The alloying addition to aluminium is to improve the physical and mechanical properties. The 6063 aluminium alloy was heat treated and the effect of precipitation hardening on the corrosion resistance and mechanical properties were studied in sea water. The specimens were aged hardened from 375K to 523K for a constant period of 6 hours and then were exposed to sea water. The effect of exposure time on thickness of the specimens, weight lost and rate of corrosion was investigated. The experimental results have shown that the percentage reduction in thickness, the weight lost and rate of corrosion were functions both of heat treatment and the exposure time. The corrosion rate is higher with under aged and peak aged specimens, but the alloy is more resistance to corrosion for over aged specimens (523K). The present investigation was carried out to explore the possibility of utilizing 6063 aluminium-magnesium-silicon alloy for desalination applications.

*Symposium [L]
14:00-15:30, Jan.9*

Synthesis and Modifications of Dendrimers on Polymer System Supported on Montmorillonite and Their Use in Organic Synthesis

El-Refaie Kenawy

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Recently, significant interest has been directed towards production of catalysts with high efficiency and high mechanical properties. Such types of catalysts are required for most petrochemical and other industrial processes. To overcome the problems associated with the conventional types of catalysts, cascade molecules (dendrimers) supported on polymer-Montmorillonite were elaborated in several directions. The polymer-clay supported dendrimers (cascade molecules) were further modified to produce phosphonium and ammonium salts. supported cascade molecules on polymer montmorillonite increased the weight efficiency of the catalyst and at the same time has advantages due to it is support to clay.

The catalytic activities of these catalytic activities of these catalysts have been examined. To the best of our knowledge, this paper is the first to describe the support of the dendrimers on clay polymer and their modification for use as catalysts in organic synthesis.

*Symposium [L]
14:00-15:30, Jan.9*

Composites and Blends

Mechanical and Thermal Properties of Pultruded Polyurethane Toughened Phenolic Resin Composites

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A novel process has been developed to toughen phenolic resin by polyurethane for fiber reinforced pultruded composites. The mechanical properties of the composites (tensile strength, flexural strength and notched Izod impact strength) approach maximum values at 10 wt% of the blocked-polyurethane content. The fabricated composites show good mechanical properties and possess low void fraction. Notched Izod impact strength of the composite (with 5 wt% polyurethane content) increases by more than 30% compared to the virgin composite.

The thermogravimetric analysis (TGA) showed that the temperature for the 5% weight loss of the phenolic/polyurethane copolymer decreases with the increasing of the polyurethane content, however, the thermal degradation temperature is still higher than 350°C. Differential scanning calorimetric analysis (DSC) showed that the on-set point of copolymer is 20°C higher than that of the virgin one. The presence of the blocked-polyurethane may hinder the polymerization of phenolic resin. The modified composite shows excellent dimensional stability and possesses good flame resistance with UL-VO and L.O.I at 50.

*Symposium [H]
10:30-12:30, Jan. 8*

Impedance and Electromagnetic Shielding of Conductive Polymer Composites

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This lecture reports the concepts and observed results on :

- 1- The AC electrical conductivity determined by measuring the dielectric constants through the impedance spectroscopy which is one of the powerful techniques normally used to characterize the electrical properties of the conductive polymer composites as we have reported in several publications in the last decade.
- 2- The insertion and reflection losses performed by the conductive polymer composites which have high capability of attenuation of the electromagnetic radiation (EMI). The shielding effectiveness of these advanced materials reaches up to 75 dB in the X-Band frequency.

However, the whole argument of the proposed lecture will shed light on the importance of the conductive polymer composites to be used as promising materials in some electronic and space applications.

*Symposium [H]
10:30-12:30, Jan. 8*

Dicyanate/Polyimide Composites With Morphology Gradient

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The morphology development and fracture behavior of dicyanate/polyimide composites having morphology gradient were analyzed. Dicyanate/polyimide composites having morphology gradient were prepared by inserting a polyimide film in the neat dicyanate resin or between the prepregs of carbon fabric impregnated by dicyanate alone, and controlling the relative rate of dissolution of polyimide and the polymerization of dicyanate by adding zinc stearate catalyst. The final morphology of the cured dicyanate revealed four different types of morphology along the thickness variation according to the polyimide composition gradient, i.e. undissolved polyimide region when the composition of polyimide was 70 wt% or more, nodular morphology when the composition of polyimide was 25~70 wt%, dual-phase morphology when the composition of polyimide was 15~20 wt% having both sea-island region and spinodal structure, and sea-island type when the composition of polyimide was about 10 wt% or less.

Such a gradient morphology was applied to the interplay of the carbon fiber reinforced composite to give high interlaminar fracture toughness. In carbon fiber composite having morphology gradient, polyimide composition in the carbon fiber which zone was low to give sea-island morphology and to provide good adhesion between carbon fiber and dicyanate matrix, and in interplay zone the polyimide composition was high to give nodular structure and to provide good fracture toughness. The composites having morphology gradient showed G_{Ic} and G_{IIc} values of 2.2 KJ/m^2 and 1.4 KJ/m^2 respectively, which showed 3~5 times of those values of unmodified carbon fiber/dicyanate composite.

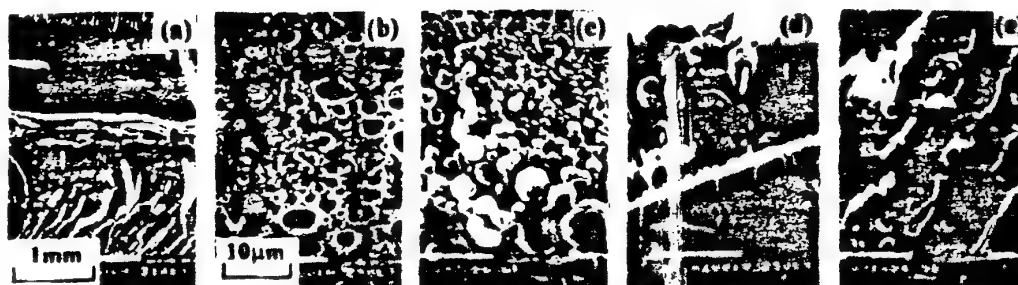


Fig. Morphologies of dicyanate/polyimide blend having morphology gradient. Overall polyimide composition = 5 wt% (a) low magnification (b) undissolved polyimide region (c) nodular structure (d) dual morphology (e) sea-island morphology.

*Symposium [H]
10:30-12:30, Jan. 8*

Reactive Extrusion Compatibilization of Poly(vinyl alcohol)-Polyolefin Blends

Lloyd M. Robeson

Air Products and Chemicals Inc, USA

Reactive blend compatibilization of highly immiscible and mechanically incompatible polymer blends has been an active area of polymer blend research for over two decades. Polyamide-polyolefin blend combinations have reached commercial status based on extrusion compatibilization. The peroxide grafting of anhydride or acid groups on the polyolefin followed by reaction with terminal amine groups of the polyamide has been well demonstrated to be a scaleable, reproducible method for mechanical compatibilization for these blends. Poly(vinyl alcohol)-polyolefin blends comprise a combination of polar, hydrophilic polymers with non-polar, hydrophobic polymers. Simple blends exhibit poor mechanical properties as expected. The extrusion grafting of maleic anhydride onto polyolefins followed by thermoplastic poly(vinyl alcohol) addition allow for anhydride-alcohol reactions and thus compatibilization.

This procedure has been shown to work with various polyolefin combined in blends with poly(vinyl alcohol) including polyethylenes, polypropylene, ethylene/propylene rubber, and ethylene copolymers. Significant improvements in tensile strength, ultimate elongation, area under the stress-strain curve (e.g. practical toughness) and notched izod strength are observed when the extrusion compatibilization procedure is employed for these blends. In essence, this procedure yields the formation of a polyolefin-poly(vinyl alcohol) graft copolymer providing surfactant characteristics to stabilize the interface between the phase separated constituents of the blend.

*Symposium [I]
14:00-15:30, Jan. 8*

X-ray Photoelectron Spectroscopic Studies of Specific Interactions in Polymer Blends and Complexes

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The formation of miscible polymer blends requires the presence of favorable intermolecular interactions as the combinatorial entropy change is too small to produce negative free energy of mixing. In certain cases when such interactions are strong, interpolymer complexation occurs as characterized by their peculiar viscosity, electrical conductivity and other properties. In addition, if the interpolymer interactions are stronger than those between either of the polymer-solvent pairs, interpolymer complexes are obtained in the form of co-precipitation from their common solvent in which both component polymers are initially soluble. Interpolymer complexes are usually obtained through strong hydrogen-bonding or ionic interactions. Fourier-transform infrared spectroscopy is the most widely used technique to study the nature and strength of intermolecular interactions between polymers in blends and complexes. We are now using X-ray photoelectron spectroscopy to study intermolecular interactions in blends and complexes involving poly(4-vinylpyridine) (P4VP_y) or poly(2-vinylpyridine) (P2VP_y). Interactions in various blends and complexes are shown by the changes in the N1s spectra. While P4VP_y and P2VP_y each shows a single nitrogen environment with a binding energy (BE) of 399.0 eV, the N1s peaks in various blends and complexes either split or become asymmetric, and can be deconvoluted into two peaks with one at 399.0 eV and another at a higher BE value. Intermolecular interactions are also shown by the O1s spectra of the other polymers. For example, in blends and complexes of poly(p-vinylphenol) (PVPh) with P4VP_y or P2VP_y, a new component O1s peak at a lower BE value appears. Other systems to be discussed include P4VP_y or P2VP_y with poly(acrylic acid) or poly(methacrylic acid).

*Symposium [I]
14:00-15:30, Jan. 8*

***In situ* Composite from Polyetherimide/Thermotropic Liquid Crystalline Polymer Blends**

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Blends of isotropic polymers and a liquid crystalline polymer have been studied for their potentials to generate *in situ* composite yielding high strength and modulus characteristics. *In situ* composite of polyetherimide (PEI, Ultem 1000 from G.E.) and thermotropic liquid crystalline polymer (TLCP, Vectra B950 from Hoechst Celanese) blend, an immiscible system, was investigated in terms of morphology, compatibility, and processing conditions.

In the binary blends, poor compatibility and weak interfacial adhesion between two phases give rather poor mechanical properties. To improve compatibility and interfacial adhesion, a compatibilizer was designed and synthesized. Polyesterimide (PEsI), as compatibilizer, was examined with each phase. It showed that PEsI was compatible with PEI but partial compatibility with LCP. The ternary blends with up to 7.5 phr compatibilizer level exhibited much improved mechanical properties giving a maximum at 1.5 phr level. The morphology by SEM confirmed that much thinner fibrils with very large aspect ratio were formed by PEsI compatibilizer for the enhanced properties. The viscosity of the *in situ* composite containing compatibilizer was higher than the binary blend indicating that there were strong imide-imide interaction and possibly trans-esterification between PEsI and PEI. The partial compatibility between PEsI and LCP also contributed to the higher viscosity of the ternary blend.

To minimize the anisotropic properties of the ternary blend, the biaxial stretching was carried out to make *in situ* composite film. The film blowing processing demonstrated high efficiency of LCP fibril formation and biaxial reinforcement. The mechanical properties of machine and transverse directions were very close proving that the film blowing technique could minimize the anisotropic properties. This film processing of the LCP fiber reinforced system suggests a new application area for the LCP dispersed blend with excellent mechanical strength.

*Symposium [I]
14:00-15:30, Jan. 8*

Miscibility and Properties of Liquid Crystalline P-hydroxybenzoic Acid Copolyester/ Polybutylene terephthalate Blends

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The copolyester of p-hydroxybenzoic acid and polyethylene terephthalate (PET) has been well known as a typical liquid crystalline polyester prepared by the acidolysis of PET with p-hydroxybenzoic acid and polycondensation through the acetate and carboxyl groups since Jackson and Kuhfus has reported its characteristics. When the liquid crystalline polymers (LCP's) are properly processed, they are to produce high degree of molecular orientation and order which should result in good mechanical strength. One of the major advantages of blending LCP's with thermoplastic polymers is that the LCP acts a processing aid. The oriented LCP domains smoothly slide past each other, thus lubricating the polymer melt and lowering the melt viscosity of the blend. In this work, we prepared thermotropic liquid crystalline copolyesters from PET with 10, 20, 30, 40, 50, 60, and 70 mole % of p-hydroxybenzoic acid by the Jackson and Kuhfus method. Blends of either of them with polybutylene terephthalate (PBT) with various compositions have been prepared by melt mixing in an intensive mixer. The miscibility and rheological and thermal properties of the p-hydroxybenzoic acid copolyester/PBT blends were investigated using a cone and plate geometry rheometer (Rheometrics Mechanical Spectrometer), differential scanning calorimeter (DSC), scanning electron microscopy and Fourier Transform Infrared Spectroscopy.

Symposium [J]
16:00-18:00, Jan. 8

Phase Diagram of PVME and Styrene-Butadiene Diblock Copolymer Blends.

Jong-Hyun Ahn, Hwan-Koo Lee, Wang-Cheol Zin

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For binary mixtures of a block copolymer and a homopolymer, they exhibit the complex phase behaviors due to the coupling of the two phase transitions; a microscopic phase separation transition of the block copolymer itself and a macroscopic segregation between the two polymers. There exists a specific interaction between the styrene-block and the added poly (vinyl methyl ether) (PVME), which is expected to give a major effect on the miscibility behavior of the blends. The purpose of this study is to obtain some experimental evidences for the miscibility behaviors in blends of PVME with a styrene-butadiene diblock copolymer (SB) by small-angle X-ray scattering (SAXS) using synchrotron radiation sources (1.5977Å in wavelength) at the 3C2 X-ray beamline, Pohang Accelerator Laboratory.

The analysis from the SAXS profile of the pure block polymer indicates that the block copolymer SB(MW=28K, wt% of styrene block=75%) has a bcc structure. As the fraction of PVME(MW=81K) in the blend, Φ_{PVME} , increases, the portion of the styrene matrix in the blend is supposed to increase because the added PVME may be solubilized only into the styrene matrix due to the specific interaction between styrene-block and PVME. Figure 1 shows D/D_0 as a function of PVME, where D_0 denotes the interdomain distance of the pure block copolymer. The predicted ratio of D/D_0 is represented by a solid line and the experimental results by the filled circles. It is found that the filled circles are very close to the line, which indicates that the added PVME is totally mixed into the styrene matrix and doesn't change the bcc structure of the block copolymer.

The light scattering experiments were performed with SB/PVME blend and the result exhibits clearly an LCST behavior at temperatures between 370K and 410K. In the previous work, we obtained the calculated phase diagram of styrene based diblock copolymer/PVME blend with lamellar structure from theoretical approach. The diagram shows an LCST phenomenon, which takes place between a homogeneous mesophase (M) and two mesophase (M1+M2) regions. M is separated into M1+M2 of different PVME fractions with increasing temperature. Therefore, SAXS can be used to determine the transition by monitoring the structural change in block copolymer and the results can be compared quantitatively with the cloud points curve. We can analogize the phase diagram of the block copolymer blends with spherical structure out of these experimental results of SAXS and light scattering.

*Symposium [J]
16:00-18:00, Jan. 8*

A New Method For Detection Of LCST-Type Phase Behavior and The Kinetics of Phase Separation Through Real Time Measurements. (TMPC/PS Blends)

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In the last few years, a great attention has been paid for the study of phase behavior of polymer blends, as well as kinetics of phase separation due to their scientific and technical importance. However, the literature data concerning the kinetics of phase separation are very little. This can be attributed to the fact that the method of detection of phase separation and its kinetics are limited and still under refinement and development. For this reason, this work deals with the development of a new method for detection of phase separation and its kinetics discussing dielectric technique. This is because the dielectric method could supply useful information about the phase behavior in earlier stages as it detects the segmental (dipolar) dynamics. Accordingly, it would be possible to evaluate the elementary steps required for the phase separation process. This new technique is applied to tetramethyl polycarbonate/polystyrene (TMPC/PS) blends to obtain a complete phase diagram. In addition, the rate constant as well as the activation energy of phase separation process have been evaluated to be in the order of $0.1-6 \times 10^{-3}$ and 55kcal/mole, respectively. It is surprising to find that although the rate constant of the phase separation process is composition dependent, the activation energy of the process is almost independent of composition. In view of the results obtained, the molecular packing and mechanism of phase separation are discussed.

*Symposium [J]
16:00-18:00, Jan. 8*

Morphology and Mechanical Properties of Nylon 6 Toughened with Waste Polyvinyl butyral Film

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Phase morphology and mechanical properties of the blends of Nylon 6 with scrap polyvinyl butyral(PVB) film have been investigated. Scanning Electron Microscope photographs of Nylon 6 with PVB revealed that the spherical PVB particles are finely and uniformly dispersed in the Nylon 6 matrix, and also appeared to be deeply embedded with PVB content in the range of 10~35 wt% PVB. Elongation at break and notched Izod impact strength of all the blends were enhanced, which implies the existence of interfacial adhesion force. In particular, the optimum PVB content for the best impact strength was founded to be in the vicinity of 30~35 wt%. In addition, Nylon 6 with 35 wt% PVB exhibited more moisture resistance than the other blend compositions. All the blends blended with up to 35 wt% PVB showed improved mechanical properties as well as those of Nylon 6 blended with SEBS as an impact modifier. Plasticied PVB film which is recycled from the process of automobile safety glass is applicable to an impact modifier of Nylon 6.

*Symposium [J]
16:00-18:00, Jan. 8*

Intercalation of Ion-Conducting Polymers in Molybdenum Disulfide

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Composites containing polymers like poly(ethylene oxide) (POE) or polyacrylonitrile (PAN) and lithium salts have proved to be useful as solid or semisolid electrolytes in lithium batteries, especially because of their properties as fast ionic conductors. Mass transport as well as ionic and electronic conductivities are also essential in the behavior of battery electrodes. In order to improve these properties in materials potentially useful as electrodes in such a kind of devices, specially in lithium-ion (rocking-chair) batteries, the synthesis of nanocomposites arising from the intercalation of these polymers into transition metal oxide and sulfides is being attempted. In this paper some studies on the synthesis and characterization of the products arising from the insertion of POE and PAN in the interlaminar spaces of molybdenum disulfide are described.

Previously reported methods, based on the activation of the MoS_2 by defoliation carried out by rapid hydrolysis of the lithiated matrix, permit the formation of POE nanocomposites. However, for the obtention of the novel nanocomposite $\text{Li}_{0.5}\text{MoS}_2(\text{PAN})_{1.0}$ described here, a new synthesis method consisting in the reduction of the matrix before the insertion of the polymer in a dry organic solvent was developed. This method is rather general might be applied also to the synthesis of the PEO derivative as well as to the insertion of other monomolecular donors as styrene and ferrocene. Contrasting with attempts to obtain these products by other methods, the procedure described here bears to greater intercalation rates, always reaching the stoichiometry expected for the insertion in the host interlaminar spaces of one monolayer of the guest.

Transport properties of the products, electrical conductivity and lithium diffusion coefficients, are described and compared with features known for similar compounds. In general, very encouraging results are found for the new family of PAN nanocomposites first reported in this work.

Research partially financed by FONDECYT, DTI-UCH, Convenio CSIC- Universidad de Chile, and Project U.E.-CONICYT CII-CT93-0330.

*Symposium [K]
10:30-12:20, Jan. 9*

Molecular Composite analysis of The Thermal Expansion Behavior of Polyoxyethylene Solid

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Semicrystalline polymers can now be regarded as a molecular version of engineering composites. The accuracy, simplicity and versatility of the molecular composite model will be highlighted. The potential of the model in predicting and analyzing the mechanical and the thermal expansion behavior of isotropic and anisotropic polyoxyethylene solids and their initial correlation with some structural and morphological parameters will be presented. It will be shown from a parametric mapping analysis, that apart from the effect of temperature the crystalline phase orientation distribution is the most crucial parameter affecting the behavior of anisotropic polyoxyethylene, followed by the volume fraction crystallinity. On the other hand the latter is of prime importance for isotropic polyoxyethylene. Furthermore, the size and geometry of the crystalline phase have negligible effects on the behavior of either isotropic or anisotropic polyoxyethylene. Good agreement is observed when comparing the theoretically predicted with the experimentally ensured thermal expansion behavior of polyoxyethylene sheets. Therefore, the molecular composite model can be reliably utilized when designing with engineering, high-performance crystalline polymers.

*Symposium [K]
10:30-12:20, Jan. 9*

On The Spectral, Thermal and Electrical Studies of 1,1, 4,4-(Buta 1,2,3- Triene) Tetrasalisoyl Tetracarbohydrazone and Its Metal Complexes

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1,1, 4,4-(buta 1,2,3- triene) tetrasalisoyl tetracarbohydrazone ($H_8BTSTCH$) was found to react with bivalent metal ions forming a plane polymeric complexes. Elemental analysis, IR and GC studies have been used to elucidate and investigate the stiochiometry and structure for the prepared samples. Science ($H_8BTSTCH$) has four hydrazone moieties, each one containing four donor groups, it coordinates with the studied metal ions as ONO system with each of the four metal ions forming tetra nuclear complexes through the phenolic oxygen, the azomethine and the carbonyl group. The thermogravimetric measurements were used to obtain the activation energy of decomposition for the ligand and its Cu (II), Co (II) and Ni (II) complexes. The dielectric spectroscopy of the ligand ($H_8BTSTCH$) and its metal complexes were measured in the temperature range 300-500 K. From the obtained results, we conclude that the activation energy derived from thermal measurements are in a good agreement with that obtained from a.c. measurements.

*Symposium [K]
10:30-12:20, Jan. 9*

Influence of the Matrix and Composition on the Dielectric Properties and the Conductivity of Composites (Polymer/Conductive Filler)

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It is known that the electrical conductivity of insulating polymers filled with conductive particles such as metal and carbon powder or fiber, discontinuously increases at certain content of the filler. Carbon black-polymer composites as conductive materials, have been the subject of extensive research. The addition of suitable type and concentration of carbon black to polymers produces compounds with different degree of conductivity.

Polymers in their pure state are excellent electrical insulators ($> 10^{12} \Omega\text{cm}$). But polymers can be modified in process to be relatively good electrical conductors ($\leq 10 \Omega\text{cm}$). Conductivity in polymer can be provided by incorporation of conductive fillers (carbon blacks, carbon fiber, and metallic powders).

This present work describes composites of systems with different type of polymers (Polypropylene, Polyethylene and Polyvinyl chloride) filled with black Acetylene (BA), in relation to the behaviour of the dielectric properties ϵ' , ϵ'' (at 9.45 GHz) and conductivity (σ) as function of composition and type of polymer.

The study shows how ϵ' , ϵ'' and evolve with filler ratio and the influence of the matrix nature on the filler distribution and than the threshold localisation.

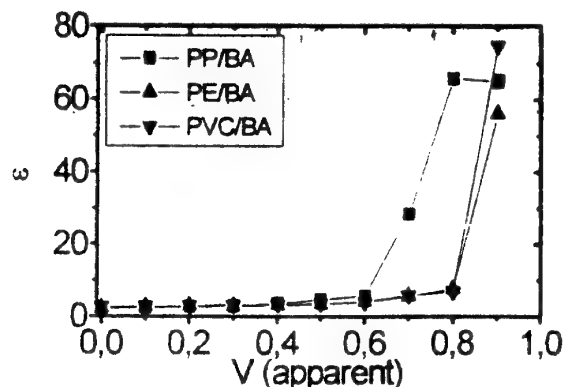


Fig. 1: Evolution of ϵ'' versus % V (apparent)

Symposium [K]
10:30-12:20, Jan. 9

Effect of Short Polyether Chains on The Ionic Conductivity of High Molecular Weight PEO-LiX Electrolytes

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Solid polymeric electrolytes formed by complexing high molecular weight poly(ethylene oxide) (PEO) with alkali metal salts are a new class of ionic conducting materials that have recently received a great deal of attention due to their potential application in solid-state batteries. Although they exhibit advantageous mechanical properties and relatively simple preparation as thin films, their conductivities at room temperature are not satisfactorily high. Since it was established that ionic conduction took place principally in the amorphous phase, several studies have been devoted to the synthesis of amorphous solid electrolytes in order to improve their electrical conductivity. One among the most promising methods is the incorporation in PEO of short chain polyethers such as poly(ethylene glycol) (PEG). Being amorphous and chemically analogous to PEO, it can therefore be expected that PEG acts as a plasticizer and causes a reduction in the degree of crystallinity. The aim of this work is to highlight the contribution of the short chain polyethers to the conduction process in PEO-based electrolytes. This was investigated following two procedures:

a) Removing low molecular weight chains from commercial PEO:

A small quantity of commercial PEO ($M_n=9.10^5$, Aldrich) was dissolved in acetonitrile and then slowly introduced in a (2/3) tetrahydrofuran - (1/3) ether mixture in order to recrystallize specifically the higher weights whereas the lower weights remain dissolved. Gel permeation chromatography analysis of the liquid phase showed that the dissolved chains had overwhelmingly a molecular weight of under 400 and represented about 7% in mass. The PEO thus purified was complexed with the $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ salt. DTA characterization revealed a considerable increase in the degree of crystallinity for the purified PEO-based electrolyte versus that prepared with the as-received PEO. Simultaneously, a.c. conductivities of the former were more than five times lower, according to the aforementioned considerations.

b) Partial substitution of PEO by PEG:

Complexes with $(\text{PEO}_{1-x}\text{PEG}_x)_8\text{LiClO}_4$ formulae, with $\text{PEO}(5.10^6)$, $\text{PEG}(1000)$ and $x=0, 0.2$ and 0.4 were prepared by conventional solution casting. In agreement with some reported results on homologous systems, we found from the DTA analysis that the degree of crystallinity decreased with the increasing of the PEG amount to yield eventually a completely amorphous $x=0.4$ compound. Moreover, the incorporation of PEG caused a slight decrease in the glass transition temperature confirming the plasticizing effect of the small molecular weight chains. These factors led to a considerable increase in ionic conductivity.

Symposium [K]
10:30-12:20, Jan. 9

Infrared Spectroscopy and Electrical Properties of Ternary Poly(Acrylic Acid) - Metal - Poly(Acrylamide)Complexes

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Fourier transform infrared spectroscopy (FTIR) and electrical measurements were used for at the characterization of the interpolymer complexation between poly(acrylic acid) (PAA) and poly(acrylamide) (PAAm) and also the ternary PAA-metal-PAAm complexes. The interpolymer complexes were prepared by adjusting the pH value of the mixture solutions at different PAA weight fractions (W_{PAA}). The ternary complexes were prepared by mixing metal chloride solutions (such as $ErCl_3$ and $LaCl_3$ with different concentration to PAA-PAAm mixtures and adjusting the pH value for different W_{PAA} . It was found that the IR spectra of the interpolymer complexes showed absorption bands at shifted positions and of different intensities from those of the parent polymers. Also, the examination of the spectra of the ternary metal-polymer complexes revealed that they depend on the nature, valency, ionic radius, and concentration of the added metal chlorides. Analysis of the electrical results showed that the electrical conductivity of the interpolymer complexes are always lower than those of PAA and PAAm which was attributed to the decrease in the mobility of the polymer chains as a result of the complexation. Also, the conductivity of the ternary metal complexes showed a dependence on the properties of the additives and were found to decrease with increasing their concentrations.

*Symposium [L]
14:00-15:40, Jan. 9*

Efficiency of Modified Phthalocyanine Compounds as Antioxidants and U.V.Stabilizers for SBR Vulcanizates

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Modified phthalocyanine compounds were prepared from the condensation of 3, 4, 9, 10 perylene tetracarboxylic acid diahydride, phthalic anhydride and urea with and without chloride salts of Fe II, Co II and Ni II, under phthalocyanine preparation conditions by the urea fusion technique.

The highly colored metal free, Fe, Co and Ni-modified phthalocyanine compounds were incorporated into styrene-butadiene rubber (SBR) mixes. The rheometric characteristics of rubber mixes were determined. The physico-mechanical properties of SBR vulcanizates were evaluated with and without exposure to thermal ageing. The obtained results revealed that Ni-modified phthalocyanine has superior efficiency as antioxidant for SBR vulcanizates, and 1 phr of that compound is the optimum concentration.

UV exposed SBR vulcanizates showed that Nickel modified phthalocyanine is a good u.v. stabilizer and 2 phr is the optimum concentration that can be significantly used in SBR. All samples subjected to uv radiation showed no significant change of colours up to 300 hrs of exposure.

Symposium [L]
14:00-15:40, Jan. 9

Synthesis, Characterization and Electrical Properties of Some Novel Phthalocyanine Polymer Analogues (Perylene Polymers)

**M. A. Abd El-Ghaffar, E. A. M. Youssef,
Noha R. El-Halawany and M. A. Ahmed***

Department of Polymers and Pigments, National Research Center, Dokki, and

** Physics Department, Cairo University, Cairo, Egypt.*

Some phthalocyanine polymer analogues (perylene polymers) mainly the metal free and its metals derivatives, namely, Cu, Ni, Co, Fe and Cr, have been prepared by the well known urea fusion technique. The prepared polymers were characterized by elemental analysis and spectrophotometric measurements e.g. IR, UV-visible, X-ray diffraction (XRD) and thermogravimetric analysis (TGA). The a.c. conductivity and the dielectric constant of the prepared perylene polymers have been investigated and studied within the temperature ranges from 300 to 523 K and frequencies from 100Hz to 100KHz. The activation energy values for conduction at high temperature ranges were calculated and showed semiconducting properties. The dielectric constant and dielectric loss were calculated and a polarization mechanism was proposed.

*Symposium [L]
14:00-15:40, Jan. 9*

Glass-Ceramic Research in Egypt

Ali Abdel-Aziz Omar

National Research Centre, Dokki, Cairo, Egypt.

The paper presents a brief summary of the main research work related to glass-ceramic science undertaken in Egypt during the last twenty five years.

The process of crystallization of glasses based on a great variety of pure silicate systems was studied. Much of the work done was also concerned with glass-ceramics based on local raw materials and industrial wastes.

The subsolidus phase relations and microstructures developed under different conditions of heat-treatment of the glasses were investigated. The effect of the different nucleation and crystallization catalysts was also evaluated.

Glass-ceramic varieties with a wide range of properties were obtained within the mineral systems: spodumene-cordierite, spondumene-enstatite, spondumene-diopside-willemite, spodumene-LiZn (LiMg) orthosilicate, celsian-diopside-anorthite, nepheline-diopside, wollastonite-diopside, plagioclase-nepheline, pyroxeneplagioclase, canasite, richterite, and many others.

*Symposium [L]
14:00-15:40, Jan. 9*

Encapsulation of Erythrocytes as Drug Carriers with Fluorescent Exogenous Agents

Mahmoud H. Abdel-Kader, R.Y.Hamzah*,
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National Institute of Laser Enhanced Science (N.I.L.E.S.), Cairo University, Egypt.

** Biotechnology Program, Arabian Gulf University, Bahrain*

Recently, attention has been focused on the investigation of new and effective routes for drug administration in order to avoid their side effects in the human body. The use of erythrocytes as drug carriers has many potential applications, including slow drug delivery to the body tissues, enhancement of drug effectiveness, and drug targeting into a specific body site. In addition erythrocytes offer a number of inherent advantages, i.e. they are naturally occurring, biodegradable, and nonimmunogenic.

This study reports on a closed loop continuous system that makes use of a plasma separator to load mammalian erythrocytes with the fluorescent exogenous agent uranin (sodium fluorescein), which is ideally suited for this study due to its desirable chemical and spectral properties. Using this system a large volume of whole blood, free of plasma protein, could be washed in a short period of time. The same apparatus can be used to homolize reversibly and reseal the erythrocytes using hypotonic and hypertonic dialysates, respectively.

In continuation of our interest in the significant applications of the system as well as to optimize the different parameters for the encapsulation process, we report our results on the encapsulation of two different mammalian erythrocytes, camel and bovine. Results indicate that the encapsulation efficiency of bovine erythrocytes was $34.0 \pm 3.0\%$ with a cell recovery rate of about 75% whereas that of camel erythrocytes was only $8.0 \pm 2.0\%$ with a cell recovery rate of about 47%. These differences demonstrate the dependency of the encapsulation process on the type of mammalian source of the erythrocytes. This closed - loop continuous flow system has the potential of processing large volumes of blood, allowing significant clinical applications.

*Symposium [L]
14:00-15:40, Jan. 9*

**Advanced Materials
from Petroleum Products**

Recent Advances in Olefin Polymerization

G. Cecchin, G. Collina, M. Galiwberti, G. Morini, L. Resconi
Montell Polyolefins. Centro Ricerche G. Natta. 44100 Ferrara, Italy

More than forty years have elapsed since the discovery of Ziegler-Natta catalysts for the polymerization of olefins, however, the activity on this research area is still continuing at an impressive pace, both in academia and industry. A first revolution in this field was brought about by the discovery of active MgCl_2 -supported TiCl_4 high yield catalysts in 1968; this discovery was the origin of an autocatalytic process that culminated with the creation of the Reactor Granule Technology (RGT) and the implementation by Himont (now Montell) of a number of extremely advanced, flexible, and environmentally friendly polymerization processes like Spheripol, Catalloy and Spherilene. Continuous research on MgCl_2 -based catalysts led Montell more recently to discover an improved generation of isospecific systems that are endowed with extremely high activity.

Today RGT, by coupling the most appropriate catalyst-process combination, is able to generate a family of olefin homo and copolymers greatly diversified in terms of mechanical, thermal, optical and rheological properties.

A new revolution in olefin polymerization was brought about in late seventies by the discovery of metallocene/methylalumoxane (MAO) catalysts. Since the beginning, Montell's approach to this strategic area of research has been of reducing to practice the potentiality of the new systems.

In particular, Montell's activity has been aimed at:

- finding out alternative cocatalysts that could replace the highly expensive MAO;
- solving the "solubility" problems of metallocene precatalysts;
- imparting to metallocenes the fundamental property they were lacking, e.g. morphology;
- developing "drop-in" and proprietary metallocene systems;
- generating new properties starting from metallocene-based polyolefins.

Based on both molecular modeling and extensive experimental research, it was possible to synthesize, using mainly MAO-free metallocenes, a series of new polyolefins such as:

- High molecular weight atactic homopolypropene (aPP) characterized by a perfectly Bernoullian distribution of stereounits along the macromolecular chain;
- Superisotactic homopolypropene endowed with both very high steric purity (mmmm > 99%) and melting point, at least for a metallocene-based PP (> 160°C);

- Superisotactic homopolypropene oligomers;
- Random polypropene copolymers with low amount of extractables;
- EPM and EPDM rubbers with narrow chemical composition distribution and tailored intra and intermolecular distribution of comonomer units;
- Narrow and broad MWD ILDPEs.

The most recent result we have achieved is a process, the "Multicatalyst Reactor Granule Technology" (MCRGT), which combines the excellent morphology control of heterogeneous catalysts with the unique properties provided by metallocenes. This technology, that can be considered as a new generation in the field of Ziegler-Natta systems, is able to generate "in situ" free-flowing, spherical "Hypersoft" polyolefin alloys.

Biodegradation of Aromatic Hydrocarbons

Riyad Y. Hamzah

Biotechnology Program, Arabian Gulf University, Bahrain

Today, there is great concern regarding the occurrence of man-made aromatic hydrocarbons in the environment. Increasing numbers and amounts of aromatic compounds are being produced industrially. In addition, aromatic compounds in natural environments as derivatives of lignin, tannins, phenolic amino acids, and other aromatic plant components also contribute to the presence of aromatics in the environment. The presence of xenobiotics in the environment may create serious public health and environmental problems.

The interest and the volume of biodegradation research have been increasing at a rapid pace and the last two decades produced a variety of reports and reviews on research describing this phenomenon, attempting to expand the range of microorganisms used for bioremediation. These investigations are searching for naturally occurring microorganisms that attack a wider range of pollutant compounds, have better pollutant degradation kinetics, and a broader range of microbial growth conditions.

Although synthetic chemicals are usually recalcitrant to biodegradation, microorganisms have evolved into an extensive range of enzymes, pathways, and control mechanisms that are responsible for catabolism of a wide variety of such compounds. The pathway by which a given compound is degraded are determined by physical, chemical, and microbiological aspects of a particular environment.

Understanding the biochemical aspects and the genetics of such microorganisms can contribute significantly to bioremediation as a tool to solve the problems of contamination. In addition to the environmental interests, biodegradation has contributed to the increasing industrial interest in the synthesis of novel chemicals.

This paper discusses the microbial utilization of aromatic hydrocarbons in terms of the formation of ring-fission substances and their degradative pathways, in addition to the induction and function of monooxygenases and dioxygenases and their role in biodegradation of aromatics. The genetics and the biochemistry of biodegradation will also be reviewed.

*Symposium [A]
13:30-15:35, Jan. 5*

Polymers Beyond 2000. A Producer's Point of View

Hamad R. Al Mohannadi, Jaafar M. Chehayber*

General Manager, Qatar Petrochemical Co.

** Head of Ldpe Marketing, Qatar Petrochemical Co*

Qatar Petrochemical Company is the first of its kind established in the GCC countries producing Ethylene and Low Density Polyethylene and now is on the verge to start the new plant allowing it to double the capacity, diversify the product range and venture into new markets.

This paper covers many subjects that a producer has to face on a continuous basis such as economics and trying to keep a healthy profit margin, widening the application to resist penetration from other polymers, advances in technology and catalysts, environmental issues and recycling and future investments in other projects of strategic nature etc

The interrelation between the various polymers in terms of feed stock and competition of application is also discussed.

*Symposium [A]
13:30-15:35, Jan.5*

Photoaccelerated Synthesis of 2-Vinylpyridines from Unsaturated Nitriles and Ethyne

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**Department of Chemistry, 342 NSM complex ,
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The heterocyclization of unsaturated nitriles and ethylene catalyzes by cyclopentadienyl 1,5-*cis,cis*-cyclooctadiene-cobalt (I) and promoted by light has been studied in toluene as solvent. Acrylonitrile and substituted acrylonitriles gave results of interest in the preparation of 2-vinylpyridines. No reaction could be observed using furmaronitrile, maleonitrile or allyl cyanide.

The (*E*)/(*Z*) - ratio of educts and products seems to be influenced by conditions in the reaction.

*Symposium [A]
13:30-15:35, Jan.5*

Recent Advances in the Etherification of FCC Olefins

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Oxygenates are being blended to increase the octane number of gasoline and to reduce toxicity of the exhaust emission. The demand for oxygenates is growing faster than any other petrochemical. This growth is due to the environmental and octane considerations. The world wide capacity of oxygenates has grown both in petrochemical plants and in the refineries. In fact, the production capacity of the refineries based ethers has increased even more compared to in the recent years. This growth of ether production emphasizes the importance of ethers in the gasoline pool. Ethers are favored over alcohols in gasoline blending because of two reasons. Ethers have a lower blending Reid vapor pressure and lower water solubility compared to alcohols. A number of commercial processes are in operation for ether production.

Methyl tertiary butyl ether (MTBE) is an important ether that is being produced since 1970 and used in US and Europe for many years as an economical octane blending component for gasoline production. MTBE is produced commercially by the reaction of methanol and isobutene catalyzed by acidic ion-exchange resins. Although MTBE is an important ether, but it is not the only ether used in gasoline blending. ETBE (Ethyl tertiary butyl ether) is another ether which has the advantage of having lower Reid vapor pressure than MTBE with comparable octane number although its oxygen contents is lower than MTBE. ETBE is produced by the reaction of ethanol and isobutene. TAME (Tertiary amyl methyl ether) is produced by the reaction of isoamylene and methanol. It possesses good characteristics as an octane booster compared to MTBE. DMPE (Diisopropyl ether) is produced by the reaction of propylene with water. It is by now the most economical ether being produced and possesses properties better than MTBE and comparable to ETBE.

The research and development is on the move to increase the yield and quality of the ethers. This effort is being put forward both on the catalysts and process development. Catalytic distillation is a process being experimented to improve the purity of the product ether as well as providing an economical option for the industry. Different types of catalysts are being explored to increase the yield of the ether and to minimize by-product formation. Modification of the existing catalysts is also being investigated to develop a better catalyst.

This paper presents an account of development in the production of oxygenates for gasoline blending. The efforts being put forward by different chemical producing companies in the catalyst and process development will be discussed. A comparison will be made among different ethers and their advantages will also be highlighted

Over-View on Manufacturing Technologies of Short Fibre Reinforced Thermoplastic Composites

M.A. Baban

Gulf Organization for Industrial Consulting, Doha, Qatar

The last three decades witnessed many advances in the field of thermoplastic composites in general and in Short Fibre Reinforced Thermoplastic (SF RTP) composites, in particular. SF RTP composites offer flexibility in design and manufacturing and can be tailor-made to suite different environments for various applications.

Compounding and shaping processes of these composites were developed and refined to maintain high reinforcing efficiency and hence to provide high performance in field applications. Such performance can be realised through the high stiffness and highly dependent on the retained fibre length and fibre orientation in the final shaped part. Compounding processes were modified to avoid the excessive damage of the reinforcing fibre during the manufacturing. Following the same line, the shaping processes such as extrusion and injection moulding were also developed and turned to control the orientation of the fibres in the final shaped parts to obtain the required mechanical properties in the preferred directions.

The current paper provides an over-view on the available technologies with the compounding of these composites and points out most influential parameters need to be considered during such process.

To evaluate, exploit and implement the recently evolved technologies in the extrusion of SF RTP pipes, the paper also provides a general guide line on the available technologies of pipe extrusion from these composites and the resources of these technologies.

In general all technologies given in this paper can be transferred and adopted within the Gulf region for industrial and domestic applications.

*Symposium [B]
16:00-17:50, Jan.5*

Low Molecular Weight Molecules Diffusion in Advanced Polymers for Food Packaging Applications

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**National Research Council, Institute of Composite Materials Technology,*

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The demand for heat stable and easily processable polymeric materials with good gas barrier properties is becoming more and more important due to the advent of new technological applications in the field of food packaging. Our attention has been focused on both a polyketone terpolymer (0.93; 0.07; 1 ethylene/propylene/carbon monoxide), and on ethylene acrylic acid ionomer in order to determine their gas and water vapor transport properties and to relate them to the polymer structure. In fact, both polymers can be envisaged as modified polyethylene, in which some of olefinic units has been replaced, at a more or less extent, with a more interactive units.

Permeability tests have been performed on both polymers at several temperatures and with different gases. Permeabilities, diffusivities and solubilities were evaluated. Their dependence on temperature was interpreted on the basis of permeation and diffusion apparent activation energies (E_P and E_D) and of heats of solution (ΔH_s). Water transport in the polyketone terpolymer was also analyzed performing both sorption and permeation experiments. Water vapor sorption tests was conducted at four temperatures (35, 45, 55 and 65°C) and at several activities.

Data obtained in the present investigation were compared to analogous results reported in literature for polyethylene in order to better highlight the effect of physical and chemical structure on the gas transportation mechanism.

*Symposium [D]
14:00-15:30, Jan.6*

New Materials Technology Transfer and Development in the ESCWA Member Countries

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Materials have always played an important role in human civilization. They play an even more important role today. In our present epoch the application of a variety of new materials in industry and agriculture has already replaced a number of traditional ones and promises to drive many more out of common usage. The displacement of traditional materials has given rise to new industrial activities and created completely novel consumption patterns.

The availability of raw materials, energy resources as well as the preponderance of eminently trainable and relatively cheap manpower resources renders several ESCWA member countries well disposed to play an eminent part in the commercial application of several new materials technologies. For these countries the question is not whether to acquire, implement and disseminate new materials capabilities. It is rather, a question of how, where and according to what criteria. Answers to such questions will have to be based on identifying areas of activity in materials technologies which are supportive of overall development efforts and which are guaranteed to play an important role in realizing long-term development objectives. A selective strategic approach is needed in which application areas are linked to resources and in which R&D and high-level training programmes may be integrated with the needs of priority segments in the industrial, agricultural and the services sectors.

The paper begins by charting some of the principal technological and economic trends in new materials development. Developments in selected new materials with special reference to areas of application of interest to the ESCWA member countries are briefly considered. The main driving forces behind R&D activities are addressed. Attempts at developing industry standards in materials technologies are also discussed. Activities which may be singled out for special treatment in future research are considered. Major developments in fabrication technologies are discussed.

The importance of downstream industrial activity based on new materials, with the concomitant technology acquisition and dissemination is discussed. This plays a crucial role in the promotion of endogenous new materials capabilities and in crossing barriers towards economics of scale in particular industrial segments. The need to conduct in-depth assessments of new materials technologies is discussed. Such assessments should encompass relevant technological issues as well as local and international economic factors.

*Symposium [D]
14:00-15:30, Jan.6*

**Advanced Materials
from Natural Products**

Chemical Modification of Lignocellulosics Using Microwave Technology

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Property enhancement of lignocellulosics by chemical modification has been studied for a long time. Most methods involve a reaction with the wood polymer hydroxyl groups. One of the most promising methods is acetylation with acetic anhydride which gives highly improved dimensional stability and improved biological resistance. The tendency of wood to absorb moisture is reduced when the hydroxyl groups of the wood polymers react with acetic anhydride forming covalently bonded acetyl groups. The equilibrium moisture content (EMC) decreases with increasing degree of acetylation. In this study the acetylation process has been performed using microwave energy as the heat source.

The use of microwave energy as a heat source in the acetylation process is likely to be most beneficial when solid wood of large radial and tangential dimensions are to be treated. In a conventional reactor the wood to be acetylated is heated from the wood surface to its interior and the heat transfer is slow. The larger the wood dimensions the more time consuming the process will be. In addition, heating the wood from the surface to its interior could lead to a lower acetylation level in the center of the wood treated, due to temperature gradients within the wood.

Microwave energy effectively heat acetic anhydride and the microwaves have a penetration depth of about 10 cm in acetic anhydride impregnated wood which means that wood samples up to dimensions of 20 by 20 cm (radial x tangential) can be evenly heated during acetylation.

When two different spruce samples, 10.5 x 9.5 x 16.0 cm; (r x t x l), were acetylated in separate experiments under the same conditions the variation in acetyl content both within and between samples were less than 2%. This implies a high degree of reproducibility in the process. Generally, a somewhat higher acetyl content was obtained in the middle of a microwave acetylated wood sample than in the outer part of it. Further studies showed that the reaction temperature (130° C) could be reached in about 10 minutes without any formation of cracks in the wood due to the fast increase in temperature.

The possibilities to use microwave energy not only for acetylation but also to get an efficient removal of excess chemicals by evaporation under vacuum has been investigated. During the vacuum step nearly 50% of the chemicals were removed after half an hour, and 70% after one hour. The remaining amount of excess chemicals will require a considerably longer vacuum period in order to be removed under the

conditions applied. Pine wood samples acetylated for 3 hours at 130° C followed by a vacuum step for two hours at 130° C, showed an acetyl content of about 19% and the content of residual chemicals was about 5% calculated on basis of dry acetylated wood. The temperature in the wood samples could be kept at 120 to 130° C even at very low contents of residual excess chemicals without any formation of hot spots, which otherwise could cause pyrolysis reaction. The results obtained imply a potential for a further removal of excess chemicals from acetylated wood by means of microwave heating.

Lignocellulosic Fiber Reinforced Thermoplastic Composite Materials

P.Gatenholm and A.Sanadi*

Chalmers University, Sweden and

** University of Wisconsin, U.S.A.*

Native cellulose fibers are the strongest and stiffest fibers available. The theoretical value for the stiffness of a single crystal of cellulose is more than 30 GPa. There has recently been a dramatic increase of interest in using cellulose fibers as fillers and reinforcement in plastic composites. Cellulose fibers offer several advantages when added to the plastic; low density, high modulus, and low price among other features. The reinforcing potential of cellulose fiber is, however, first fully utilized when stress is transferred efficiently at the fiber/plastic interface. Strong interfacial adhesion is required for proving efficient stress transfer. In this study the interfacial adhesion and dispersion of cellulose fibers in cellulose-plastic composites were improved by surface engineering of cellulose fibers. Molecular interactions between cellulose and plastic matrix were provided by plasma and ozone treatment of surfaces of cellulose fibers, surface decoration with tailor-made compatibilizers and by promoting transcytallization at the interface.

*Symposium [F]
10:30-12:30, Jan. 7*

Applications of Natural Fibers in Automotive Industry in Brazil -Thermoforming Process

A. Leão and N. Tavares*

Professor of UNSEP - São Paulo State University -

F.C.A. - Campus of Botucatu. SP Brazil

** Toro Ind. & Com. Ltd. - Diadema, SP, Brazil*

Several natural fibers, such as sisal, jute, ramie, coir, curauá, hemp, flax and sugar cane bagasse were used in the thermoforming process for automotive applications. The variables were ratio polypropylene/natural fibers, gramature, temperature of pressing, time of pressing, moisture content of the mats, denier of the polypropylene, melting flow index of the propylene, and the specie of the fibers. The parameters evaluated were the modulus of rupture and elasticity, impact resistance, Izod-notched and unnotched, dimensional stability, fire resistance and the creep resistance.

The results were considered under the end-use perspective, with different properties for each part to be developed. The life cycle assessment has been used for evaluation of the best option under recycling and environmental impact, compared with man-made materials. The statistical analysis were carried on using SAS, with test F and Duncan to compare the treatments. The results showed that the main variable was the fiber type, with curaua resulting in the best composite. In the second group we found the sisal and jute; ramie was the third, followed by the others. Later on, this work will be continued with extrusion and injection methods, using natural fibers and plasma treated raw materials.

*Symposium [G]
16:00-18:00, Jan.7*

Educational Needs and Trends

Polymer Education and Research in The Mediterranean Basin and The Middle East: Trends and Needs at The Eve of The 21st Century

D. Benachour

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All over the world, new trends and needs in the field of polymer educational programs as well as applied and basic research topics, are appearing. This is due to several factors, the major ones being: new and strong policies to protect the environment, globalization and regionalization of the world market, internationalization of sciences and technologies, opening of new opportunities induced by development needs as well as by new economical politics (examples of Algeria and Egypt) updating of academic curricula...At the eve of the 21st century, these changes must be taken into account by all concerned parties: universities, industries, economists...

This presentation will be divided in two parts. In the first one, we will raise some questions such as:

- is there a need for changes of the academic curricula? For instance, is it time to have a Bachelor of Engineering" different from a "Bachelor of Science"? Is a Ph.D. degree necessary to enter the industrial world? At what level should be introduced "polymers"?... This type of question is being asked all over the world, from America to the Far-East, from Europe to Africa, from Australia to Central Asia...Some responses are thought over. new academia curricula, more collaboration between universities and industries, joint research programs As illustrations of theses changes, we could mention the emphasis on "environmental friendly" polymers and the attention given to "conducting" polymers in the last years!

In the second part of our presentation, we will discuss some trends and needs that are appearing in some countries (Algeria and Egypt would be good examples). We will emphasize on those related to:

- changes induced by new economical politics (disappearance of "socialist" economics all over the world)
- the internationalization of economics and markets: appearance of new "world markets" (blocs such as U.S.A.-Canada-Mexico, Western Europe, Japan and the Far-East...)
- the rapid growth of communication technologies: a multi-media system is becoming a common instrument of work, networking is now a must (electronic mail is already spreading all over the world) and a wireless phone is no longer a curiosity...

- the fact that countries having regional needs are getting more and more into "Networks" (MEDNET for instance) and involved into more joint programs (MED-CAMPUS, for example) This is resulting into new trends and opportunities for industries as well as universities.

These new trends and needs in polymer education and research will be discussed in more details for two countries: Algeria (as a Mediterranean country) and Egypt (as the heart of the Middle East)

At the eve of the 21st century, it is time for every person and every country, involved somehow in the field of polymer education and research, to take into consideration the new trends and needs that are appealing. Such needs and trends will reshape, slowly but surely, not only related academia curricula but also industrial profiles and markets. This has already started in some countries, others (such as those of the Mediterranean basin and the Middle East) must catch up...

A New Approach Materials Education for Emerging Countries

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The advent of computers into education saw us improving our presentations with Harvard Graphics and Power Point, etc. As compilers, using visual basic and C++ improve, so has the presentation on the computer screen. Screens now are interactive with the student and provide large amounts of visually attractive information.

The standard of attractiveness is improving dramatically in the short term. The materials computer book for undergraduates in Britain is a very useful first. The internet courses from Louisiana State University on Polymer Science are being added to daily, the course from Carrier on Polymeric Materials and Processing in Canada also pertains.

Of more interest for the emerging countries is

- to introduce material concepts and terminology in the senior school science program;
- to strengthen the interaction between the student and his course material by making it more interesting;
- to make reasonably priced or free packages available to teachers and students where similar material is unavailable in these poorer nations;
- to use the same courses on the shop floor of industry to boost awareness and provide training and re-education programs, even where there is generally even language skill difficulty.

With multimedia benchtop computers you can now have a conversation with the computer and not just words but accurate digital pronunciation is available from the computer while the computer points from the mouse you are using to sections of a picture.

The first beginnings in this area was initiated at the University of Illinois by Prof. Park in Prof. Economy's Group where a number of lectures on materials have been designed for the senior school curriculum.

Our experiences with this curriculum and our experiences with our own curricula material will be discussed against the background of the needs as perceived in Africa. The lecture will be a multi-media lecture.

*Symposium [C]
10:30-12.05, Jan 6*

Benefits of Capstone Design Courses in Materials Education

R. W. Heckel, C. L. Nassaralla, J. Pilling and M. R. Plichta

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The many educational benefits of capstone design courses are being realized in the rapidly growing number of universities whose engineering departments are insetting such courses into their curricula. As a pan of a complete revision of the upper division curriculum, our department added a senior year (three academic quarters at four credits per quarter) capstone course entitled "Materials Processing and Design" (MP&D). We are currently in the midst of the third offering of this course.

MP&D is team taught with designated faculty responsibilities. Although some traditional academic format is included in the first quarter, the course emphasizes student teamwork with one or two faculty advising each group. Typically, there have been seven to nine student teams with four or five students per team. The hallmark of this course is the association of each team with an Industrial Participant (IP). The IPs offer topics for student selection at the end of the junior year, teams are constituted by the faculty on the basis of topic interest, topics are subdivided by students into several projects in the first quarter of the senior year and a proposal for team activity is written and presented orally at the end of the first quarter. The second and third quarters are devoted entirely to team activities as proposed; faculty advisors meet with teams at least weekly; IPs are also involved in various forms of communication and in sponsoring plant trips. The highpoints of the second and third quarters are the reports and presentations at the end of each term. The presentations are in a formal setting; the students organize and chair the sessions; the audience includes the IPs, faculty from the engineering college and administration from the University. Most students also attend national professional society meetings at various times throughout the year.

The faculty are enthusiastic about the very positive impact of this capstan course and the students and IPs have responded very favorably to it. The IPs add engineering reality to the course. Faculty are welcomed by students as resource persons and are not viewed as being in the traditional "teach and evaluate" mode since there are no examinations throughout MP&D. Further, students perceive the course as being a very important link between their education and their professional careers. This presentation will focus on the nature of the MP&D, the IPs, the topics selected by the students, successful topic outcomes, educational successes and several areas which we are trying to improve.

*Symposium [F]
10:30-12.00, Jan.7*

Multi-Institutional Collaboration on Educational Reform: The Case of The Engineering Education Coalitions

Grechen Kalonji

*Kyocera Professor of Materials Science
University of Washington*

Beginning in 1990, the National Science Foundation (NSF) in the United States launched an ambitious, imaginative and qualitatively new approach to engineering education reform with the creation of the Engineering Education Coalitions (EEC) program. The crux of the EEC approach is that, rather than funding curricular reform projects at **individual** schools, the NSF would support the creation of **cross-institutional entities**, called coalitions, in which multiple institutions pursue a common strategy toward the reform of engineering education and its infrastructure. All of the coalitions share the same basic mandate, which incorporates transforming curricular content and pedagogy, creating new modes of interaction with industry and the broader education community, changing faculty roles and rewards, and making engineering education a more effective and attractive option for a broader cross-section of youth, especially for women and underrepresented minorities. The funding of each coalition amounts up to the value of \$ 3 millions from the NSF, over a five year period, with continued funding contingent upon the demonstration not only of concrete accomplishment at each member school, but of effective collaboration among schools towards the coalition's objectives. Currently, there are eight coalitions nationally, incorporating 58 educational institutions. Of the bachelor's degrees awarded in engineering in the US in 1992-1993, approximately 34% were from schools which are members of coalitions, so the potential for wide spread impact is high. In this paper, we offer an overview of the coalitions program as a whole, highlighting the strengths and weaknesses of this novel approach to curricular reform. We focus on the programs of one of the first-to-be-funded of the coalitions, ECSRL, which has recently been renewed by the NSF for the second five years of funding and with which the author has the greatest familiarity. Through a description of what worked and didn't in ECSRL's first five years, and through a description of our programs for the renewal period, we hope to elucidate some general principles for operating in such a complex, multi-institutional areas.

*Symposium [F]
10:30-12.00, Jan. 7*

Novel Processing Methods

Processing and Characterization of Nano-Structured Ceramic / Organic Hybrids

Shin-ichi Hirano

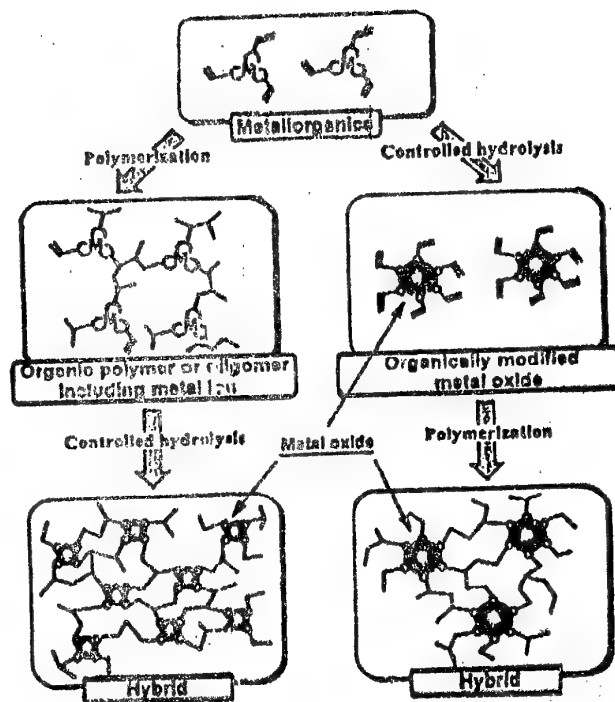
Department of Applied Chemistry, Nagoya University, Nagoya, Japan

Nano-structured ceramic particles / organic hybrid have been receiving great attention in emerging era. The conventional blending of fine particles into polymer or oligomer causes the particle agglomeration, leading to the degradation of the desired properties. The newly developed method is based on the nucleation and growth control of crystalline oxide particles in organic matrix through the reaction control of metallorganic precursor with combination of the hydrolysis and the polymerization below 100°C.

The nano-sized magnetite particles / polymer hybrid was synthesized at around room temperature from designed metallorganic molecules with vinyl group. The hybrid exhibits the interesting features of superparamagnetics and quantum effect. The hybrids can be processed in optically transparent thin films.

Examples are the in situ processings of nano-sized crystalline iron oxide/ BaTiO_3 / PbTiO_3 / MgAl_2O_3 / organic hybrids derived from metallorganic precursors susceptible to hydrolysis and polymerization. This novel method is a promising key for introducing new family materials.

(This work has partly been sponsored by NEDO).



Symposium [E]
16:00-18:05, Jan.6

Smart Thin Films via The Sol-Gel Route

J. Livage

Chimie de la Matière Condensée.

Université Pierre et Marie Curie - Paris - France

Transition metal oxides thin films (V_2O_5 , MoO_3 , WO_3) have been deposited via the sol-gel route. They exhibit ionic and electronic properties and are specially well designed for the realization of thin film devices. This paper discusses some applications of these sol-gel deposited thin films.

Vanadium oxide sols and gels are made of long ribbon-like oxide particles which macroscopically orient in the same direction like lyotropic nematic liquid crystals. Anisotropic thin films can be deposited from these sols. They exhibit improved electrochemical properties as reversible cathodes for lithium batteries. Amorphous oxopolymers are formed via the controlled hydrolysis of vanadium alkoxides. They allow the deposition of optically transparent thin films that exhibit interesting electrochromic properties and turn reversibly from yellow to green upon electrochemical reduction. Moreover these alkoxide derived films can be easily reduced into vanadium dioxide. These VO_2 thin films exhibit thermochromic properties and could be used as optical switches in the interface. The transition temperature of these VO_2 films can be modified by doping the vanadium oxide with other cations. Interesting results have been obtained with Ti doped VO_2 films.

Tungsten oxide thin films are known to exhibit electrochromic properties. They reversibly turn from colorless to blue upon electrochemical reduction. Such films can be deposited from aqueous solutions of tungstic acid. However condensation is very fast and the precipitation occurs within few hours. These sols can be stabilized via the complexation of molecular precursors by peroxide ligands. Longer life time and better reversibility have been obtained with Ti doped WO_3 thin films.

The sol-gel process is highly amenable to incorporating organic or biological species into oxide matrices. these hybrid nanocomposites open new possibilities for optical devices or chemical sensors.

*Symposium [E]
16:00-18:05, Jan.6*

Synthesis and Processing of Nano-Scale Materials Through Chemistry

Helmut Schmidt

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Nanoparticles have become of interest for materials science since more than ten years now. Despite the high potential, however, the availability of nanoparticles for making materials is still low. For this reason, a chemical synthesis process has been developed using interfacial thermodynamics for controlling growth and aggregation. During the nucleation and growth process in the presence of molecules interacting with the surface, particle size and surface reactivities have been controlled. The generation of nanoparticles in the presence of polymerizable monomers leads to liquid systems which can be cured to nanocomposites. Based on these basic findings, a variety of materials in form of nanocomposites has been developed. Examples are hard coatings and polycarbonate for automotive glazing, superparamagnetic iron oxide particles coated with functional groups for cancer therapy or low-loss waveguiding materials. All materials are using sol-gel techniques as preparation principles and surface modification with functional silanes to stop particle-to-particle interactions, to avoid agglomeration, and to generate functional groups as a link to the matrix. Synthesis principles, materials characterization and examples for the application of these materials are given.

*Symposium [E]
16:00-18:05, Jan.6*

Microporous Mixed Oxides a Sol-Gel Approach to The Design of New Heterogeneous Catalysts

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Due to the increasing economical demands and ecological on new chemical processes there is a growing need for more selective new catalysts. Amorphous microporous mixed oxides with homogeneous elemental distribution and a narrow pore-size distribution can be synthesized in the absence of organic templating agents by a specific sol-gel process. The microporosity of the materials is dependent on experimental conditions of the sol-gel procedure as well as the drying and calcination process. The elemental composition of the mixed oxides can be controlled by acidic copolycondensation of suitable hydrolysable precursors. Copolycondensation with precursors containing one nonhydrolysable alkyl group, such as $\text{MeSi}(\text{OEt})_3$, provides control over the surface polarity of the final catalyst. These materials are characterized by a type I adsorption isotherm. Rheological studies show a simultaneous increase in elasticity and viscosity under these sol-gel conditions, indicative of a linear chain growth in the polycondensation process.

These microporous materials show shape selective catalysis as well as selective redox catalysis. Hydrophilic materials are effective oxidation catalysts with organic hydroperoxides while the hydrophobic mixed oxides can utilize aqueous hydrogen peroxide in selective oxidation reactions.

Microporous membranes with pores $< 1\text{nm}$ and a narrow pore size distribution can be prepared by the combination of dip-coating and above mentioned sol-gel procedure. Such membranes show superior separation properties with rejection limits at molecular weights of 180 and smaller. If catalytically active, these membranes can be used as catalyst membranes, which combine separation properties with catalytic activity in a single operation. Several examples will be presented, which document the advantage of such a new reactor technology over conventional process conditions.

*Symposium [J]
16:00-18:00, Jan.8*

Application of Sol-Gel Processing in The Manufacture of Functional Optical Coatings

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As a versatile preparation procedure, the sol-gel technology provides a good opportunity to the production of a large variety of oxide and organic-inorganic hybrids in various shapes such as coatings, powders, fibers and monoliths from liquid precursor materials. The unique advantages of sol-gel process, especially low processing temperature and wet chemical environment, allow the introduction of both inorganic salts or ions and organic molecules into the inorganic network, leading to the acquirement of the targeted properties.

Optical coatings with certain function or functions prepared by sol-gel method are a rapidly expanding research field. In the present work, we report on the application of sol-gel process in the production of functional optical coatings, including moisture-resistant (MR) coatings, anti-reflective (AR) coatings, photo-chromic (PR) coatings and phase change (PC) coatings.

MR coatings prepared from organically modified silanes show excellent protective function to phosphor laser glass and potassium dihydrogen phosphate (KDP) crystals, which are used in our Laser Inertia Confinement Fusion (LICF) system. The laser induced damage threshold (LIDT) of the glass and crystals has been greatly increased up to 4.9-5.2 J/cm² i.r.t 250 ps pulse at 1064 nm wavelength. The porous silica AR coatings on laser glass surfaces resulted in a 6.5-7.0% increase in the transmission at 1064 nm and 532 nm wavelengths. Sol-gel derived PR coatings by doping spirooxazine into organic-inorganic hybrid matrices showed encouraging performance: the photochromic response and the thermal decolouration rate of the coatings are similar to those of the dye-in-ethanol solution, while the photostability of the dye is improved by a factor of >50 in comparison to the dye solution. Vanadium dioxide PC coatings were also successfully prepared via sol-gel processing and showed significant thermochromic effect due to reversible metal-semiconductor phase change. Effects of dopants on the PC behaviour were also investigated. Both the PR and PC coatings may be good candidates as recording layers in optical discs.

*Symposium [J]
16:00-18:00, Jan.8*

Materials for Separation Technology

Application of Polymer Membranes to Alcohol Concentration in Fermentation of Biomass

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It is required that aqueous alcohol solutions obtained from a fermentation method of biomass are concentrated and separated by some methods to use concentrated alcohol as fuels. There are many techniques for concentration and separation of aqueous alcohol solutions. Membrane separation techniques are worth noticing in the point of view of energy saving. In this paper, two types of polymer membranes such as water-permselective and ethanol-permselective membranes were applied to the concentration and separation for aqueous ethanol solutions and permselectivities through their membranes in pervaporation (PV), evapomeation (EV) and temperature difference controlling evapomeation (TDEV) are discussed from the point of view of chemically and physically structural characteristics of their membranes.

Water-permselective membranes

Hydrophilic polymer membranes such as chitosan and alginic acid in the permeation of aqueous ethanol solutions were not only water-permselective, but also hydrophobic polymer membranes such as polystyrene and poly (vinyl chloride). The water-permselectivity among these hydrophilic and hydrophobic membranes can be explained by solution-diffusion model.

The water-permselectivity, permeation rate and degree of swelling of chitosan membranes cross-linked with glutaraldehyde for vapors of aqueous ethanol solutions in EV were greater than those of the chitosan membrane. These results can be discussed from changes of chemical and physical structure of the glutaraldehyde cross-linked chitosan membranes based on the degree of hydrogen bond of the membranes.

Ethanol-permselective membranes

The water- and ethanol-permselectivity in PV of graft copolymer (MMA-g-DMS) membranes prepared from copolymers of methyl methacrylate (MMA) and oligodimethylsiloxane (DMS) macromonomer significantly depended on the DMS content. These different permselectivities can be explained by a discontinuity and continuity of the DMS phase in the microphase separation of the MMA-g-DMS membranes.

Poly [1-(trimethylsilyl)-1-propyne] (PTMSP) membranes modified with polymer additives contained a fluorine component showed higher ethanol-permselectivity and higher permeation rate in PV than those of the PTMSP membrane. These high membrane performance for the ethanol-permselectivity can be understood by a high water-repellency of the surface of modified PTMSP membranes.

As poly(dimethylsiloxane) and PTMSP membranes which showed typical ethanol-permselectivity in PV were applied to TDEV, they had higher ethanol-permselectivity than in PV. These results can be explained by both a high affinity of ethanol molecules to the membranes and a high aggregation of water molecules in TDEV.

Transport of Multicomponent Gas Mixtures Through Polymeric Membranes

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In recent years many different types of polymeric membranes, both asymmetric and composite, have been developed for gas separation applications including oxygen enrichment in air, hydrogen recovery from hydrocarbons refinery operations, and natural gas sweetening (H_2S and CO_2 separation from hydrocarbons). Depending on the structure of the membrane and the nature of the gases to be separated, different mechanisms describe permeability and selectivity of the membranes. Both asymmetric and composite membranes have two layers of dense and porous structure. In the dense layer solution - diffusion mechanism and in the porous layer pore flow mechanism describe the behavior of the membranes. In this paper a combination of these two mechanisms is presented for prediction of separation and permeability of the membranes. It is shown that in the dense and nonporous layer of the rubbery membranes absorption based on Henry's law and diffusion by Ficks' law describe transport of gases through the membrane, whereas for glassy membranes transport through the dense layer of the membranes is described by Henry's law for absorption, Langmuir isotherms for adsorption and Fick's law for diffusion. In the porous layer, Knudsen - Poiseuille flows are used to describe transport of gases through the membranes. Pore characteristics and physicochemical properties of gases are used to predict membranes permeability and selectivity. The predictions of the models are compared with the data reported in the literature for both binary and multicomponent gas mixtures.

*Symposium [I]
14:00-15:40, Jan. 8*

New Materials for Analytical Separations Using Capillary Columns

**Luis A. Colón, Yong Guo, Adam M. Fermier, Kimberly J. Reynolds, Rafael
Alicia-Maldonado, and Joseph A. Gardella, Jr.**

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In recent years we have seen a general trend toward miniaturization of the technology used to perform analytical separations. This is reflected by the development of a variety of capillary separation techniques (CST), particularly for liquid separations. In these techniques, separation is achieved in capillary tubes (open tubes or packed columns), offering numerous and very attractive advantages compared with existing technology. For example, CSTs provide increased mass sensitivity, high efficiency, low solvent consumption, low operational cost, and the capability of handling extremely low volume samples ($<1\mu\text{L}$). Of particular interest is the study and further development of capillary electrochromatography (CEC). In this technique, an electric field is used to drive the solvent through the separation capillary column, via electroosmosis, without the use of a pressure gradient. One critical aspect of this emerging technology is the fabrication of suitable separation materials inside the capillary columns. It is also desirable to fabricate materials with characteristics that are superior to those currently available.

We are studying new approaches to fabricate suitable materials for CST. In one approach, we use sol-gel technology to fabricate an organic-silica material inside capillary columns. The material is cast as a thin film onto the inner walls of the capillaries, which are then used to perform separations. A sol-gel solution containing tetraethoxysilane (TEOS) and *n*-octyltriethoxysilane ($\text{C}_8\text{-TEOS}$), among others, is used to prepare the organic-glass film inside the capillary columns ($5\text{-}75\text{ }\mu\text{m i.d.}$). The C_8 moiety gives the necessary organic character to the silica-glass support to serve as the stationary phase for reversed-phase liquid chromatography. The fabricated material has been tested in open tubular electrochromatography (OTEC) and open tubular liquid chromatography. The capillary columns coated with the sol-gel material have been shown to possess excellent pH stability and outperformed columns prepared by conventional procedures.

In a second approach, we are exploring the possibility of using polytetrafluoroethylene (Teflon) as a support material for CST. The high chemical resistivity of polytetrafluoroethylene is a very attractive characteristic of this polymeric material. However, the low chemical reactivity of Teflon® has also hindered its use as a support for a stationary phase. We are using a radio frequency glow discharge (RFGD) plasma to modify the surface of polytetrafluoroethylene particles (3-5 μm i.d.). The modification consists of incorporating hydroxyl groups onto the polymer surface. This provides a site to chemically attach a stationary phase for use in liquid chromatography. This material is being tested for CEC.

Effects of Plasma Treatment on Gas Permeation Properties of 6FDA-p-TeMPD Polyimide Membrane

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**Samcheon Gas Industry*

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The surface of 6FDA-p-TeMPD polyimide, which was synthesized by chemical imidization, was modified by plasma treatment using Ar, CF₄ and C₃F₆. The effects of plasma conditions such as initial pressure, discharge power, gas flow rate and treatment time on gas permeation properties of the modified membrane were investigated in detail.

The XPS spectra showed that the surface of the Ar plasma-treated membrane was fairly different from that of untreated membrane due to the ablation of carbon moieties and the reorientation of fluorine moieties in the polymer surface. The O₂/N₂ selectivity and the O₂ permeability were 4.2 and 67 barrer, respectively, under the conditions of 0.12 Torr initial pressure, 100 W discharge power, 4.7 sccm Ar flow and 10 min treatment time.

The effects of the CF₄ plasma treatment on the gas permeation properties of the membrane exhibited the similar tendency to those of Ar plasma-treated membrane. The selectivity of the CF₄ plasma-treated membrane, however, was smaller than that of Ar plasma-treated membrane. In the case of C₃F₆ plasma polymerization treatment, the O₂/N₂ selectivity and the O₂ permeability of the film were influenced mostly by discharge power and monomer flow rate, and reached 5.0 and 60 barrer, respectively, under the conditions of 0.12 Torr initial pressure, 60 W discharge power, 2.5 sccm monomer flow rate and 10 min treatment time.

*Symposium [I]
14:00-15:40, Jan. 8*

Group Contribution Methodology for Predicting The Permeability and Permselectivities of Aromatic Polymers

Lloyd M. Robeson

Air Products and Chemical, Inc., USA

Membrane separation has evolved into an important separation method for various gas mixtures (e.g. O₂/N₂). Aromatic polymers such as polysulfones, polycarbonates, and polyimides are commercially employed for many of these separations. The ability to predict the permeability and permselectivity from polymeric structure units has been a long term goal of this area of technology. The approach noted in this presentation involves the use of the basic equation:

$$\ln P = \sum_{i=1}^n \phi_i \ln P_i$$

where ϕ_i = volume fraction of a structural unit i and P_i = the permeability contribution of the structural unit. The volume fraction contributions of the structural units were calculated using existing computer software. Experimental permeability data were compiled into a series of equations and solved by least squares fit to yield the respective values of P_i .

This procedure has been applied to 24 structural units with 65 polymers comprising the data base. The polymer families included in this initial analysis were polycarbonates, polyarylates, polysulfones, poly (aryl ethers) and poly (aryl ketones). Excellent agreement for the prediction of O₂, N₂ and He permeability was observed with good agreement between experimental and predicted results for permselectivities of the gas pairs. This procedure provides a quantitative assessment of the structure / permeability characteristics of the various structural units comprising polymers of interest. Further refinements of this technique involving additional polymeric families (e.g. polyimides and polyamides) in addition to an expanded database will allow for a powerful predictive tool for this important technology.

*Symposium [J]
16:00-18:30, Jan. 8*

The Highest Gas Permeable Membranes of Poly[l-(trimethylsilyl)-1-propyne] - Their Gas Permeability and Modification -

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The highest gas permeable membrane of all both natural and synthetic membranes is poly[l-(trimethylsilyl)-1-propyne] (PMSP, PTMSP) membranes. Its oxygen permeability, for example, is about 20 times higher than that of poly(dimethylsiloxane) (PDMS) membrane at room temperature, though the selectivity is very low. The reason of such high gas permeability is that the PMSP membrane has the highest diffusion coefficient and also very high solubility of gas. The both high diffusion and solubility coefficients of gases come from its large intersegmental distances and at the same time large excess free volume in the PMSP membrane. However the biggest problem of the PMSP membrane is a decrease in its gas permeability with course of aging and absorption of non-volatile organic vapors. The following items were investigated:

- 1) Aging phenomena. The aging process was based on relaxation of the unrelaxed volume. PMSP is the typical glassy polymer. Therefore, using the dual-mode sorption and transport model, the unrelaxed domain in the membrane, namely the Langmuir's domain, was discussed, during aging, CH , which is the hole saturation constant in the Langmuir's domain, decreased. The effect of the physical aging on the molecular chain motion was also studied in terms of the spin-lattice relaxation times (T_1) using solid state NMR analysis. During aging, the chain packing increased i.e. the unrelaxed volume decreased and the T_1 values of the backbone chains increased. However, those of the side chain carbons didn't change. The gas permeability decreased also by the thermal hysteresis of PMSP membrane. The gas permeability coefficients at 30°C strongly depended on the highest thermal hysteresis which the membranes have experienced.- Another reason of the decrease of the gas permeability is the blockade-effect caused by the absorption of non-volatile oil vapor, because the PMSP membrane have a strong affinity for hydrocarbons.
- 2) Modification of PMSP and its membrane for stabilization. Several modification methods have been investigated, such as halogenation, the high energy irradiation. Here, the modification of the filling of PDMS oligomer, copolymerization with phenylpropyne (PP) and the blending of its polymer (PPP) to stabilize the gas permeability by controlling the larger size of excess volume is discussed. The addition of a small percent of PDMS oligomer, co-monomer PP and PPP was enough for the stabilization.

- 3) Modifications for the effective separation membrane with both high gas and liquid permeabilities and selectivity. Making the best of the highest gas permeable properties of PMSP membrane, the following modifications were carried out:
- a) The sour gas separation by the addition of affinity chemicals such as 3-methylsulfolane to SO_2 . The separation factor, SO_2/N_2 , increased remarkably without so much sacrifice permeability coefficients.
 - b) The separation of halogenated hydrocarbons, such as trihalomethane in the diluted aqueous solution by the addition of fluoroalkylmethacrylates followed by the irradiation of gamma ray in nitrogen atmosphere. By this modification, both flux and the separation factor, chloroform/water increased. These modified PMSP membranes are useful for the environmental protection.

Oxygen Transport through Electronically Conductive Polyaniline

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Polyaniline has been attracted considerable attention because it is electronically conductive, and thermally stable and soluble in organic solvent such as NMP. Recently, a remarkable oxygen permselectivity over nitrogen has been observed in the polyaniline membrane when doped with appropriate protonic acids. It has been, thus, paid much attention as a potential gas separation membrane material. The reported transport properties strongly depended upon the doping conditions and their level. Upon doping polyaniline with protonic acids, its physical structure will be changed and the polaron (radical cation) will also be generated. Here, the transport properties of oxygen will be attempted to interpret in terms of the facilitated transport and the structure change upon doping with HCl.

When the doped polyaniline was exposed to oxygen, the EPR line intensity decreased with oxygen exposure time. This manifested that the polaron reacted with molecular oxygen and lost its paramagnetic property. The line intensity was also measured while evacuating. Its initial intensity was completely recovered, which demonstrates the reversible interaction. Therefore, the magnetic interaction between oxygen and polaron is found to be *specific and reversible*. Therefore, the polaron can act as an oxygen carrier for facilitated oxygen transport. In facilitated transport, the total effective permeability is summation of the Fickian permeation and carrier-mediated permeation, giving the increased effective permeability with the carrier concentration. It was found here that the polaron, an oxygen carrier, increased initially, but the oxygen permeability decreased with doping level, demonstrating no correlation between the permeability and the carrier concentration. Therefore, it was concluded that the facilitated transport might occur, but its contribution to the total permeation was minor.

When polyaniline is doped with protonic acid, densification can occur. The *d*-spacing to characterize densification was obtained from X-ray diffractograms of differently doped polyanilines. If we assume that the free volume is proportional to the cube of the *d*-spacing. The free volume theory for diffusion can be written by

$$D = D^0 \exp(-B_d / d_3) .$$

We found a fairly linear relationship between $\ln(D)$ and $1/d_3$, together with polyimide, polysulfone and polycarbonate. This result suggests that the permeation behavior is primarily controlled by structure change upon doping. This is also consistent with the high separation factor for pairs of gas mixture such as H_2 , CO_2 , CH_4 etc., which does not have any specific interaction with the polaron.

Preparation of Silica-based Membrane for H₂ Separation by Multi-step Pore Modification

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Inorganic membrane for gas separation has been developed for the past two decades because of its potential applicability due to its temperature resistance, high permeability and high energy efficiency. Porous inorganic membranes can be usually prepared by sol-gel process, chemical vapor deposition and pyrolysis method. In sol-gel process, silica and γ -alumina sols are frequently used since their gels have high porosity and high surface area. Silica can be also deposited on support by chemical vapor deposition. In this research, sol-gel and CVD methods were employed sequentially. First, *in-situ* silica sol-gel process was conducted onto the α -alumina support that has an asymmetric structure and average pore size of 80 nm in its dense layer. In this step, TEOS and acidic alcohol-water mixture diffuse into the inside of the porous support countercurrently. Silica sols are formed in the intermediate layer of the support, and simultaneously experience partial gelation. After dense silica gels are formed inside of the support, palladium catalysts are deposited to enhance hydrogen selectivity by Soaking and Vapor Deposition (SVD) procedure. Finally, silica sols are coated on both the end regions of the membrane to prevent channeling through cracks. The membrane prepared by the present multi-step pore modification method shows a moderate hydrogen selectivity and high hydrogen permeability (about 10^{-6} cm³(STP) / m² sec kPa) at high temperature and at high transmembrane pressure ($\Delta P = 40$ psi).

Symposium [J]
16:00-18:30, Jan. 8

Pervaporation of Alcohols by Using Tubular Type Cellulose Acetate and Polysulphone Membrane with Ceramic Support

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The dehydration of ethanol and isopropanol was performed by using the tubular type membrane, which was prepared by dip-coating and rotation-drying technique. The polymers coated on porous ceramic support were cellulose acetate and polysulphone. The active layer of polymer film was formed either on inner or on outer surface of the ceramic support. The thickness of the intermediate layer between active layer and ceramic support is estimated to be about 1 μ m. The surface of the intermediate layer contacted with the ceramic support is very rough, which is caused by permeation of polymer into the pore of the ceramic support. It seems to strengthen the durability of the polymer layer.

The characteristics of pervaporation with these membranes were investigated for ethanol/water and isopropanol/water system. The effects of operating temperature and concentration of retentate on selectivity and flux were discussed. In case of cellulose acetate coated membrane, the selectivities of water were 4-11 in ethanol/water system and 8-240 in isopropanol/water system. As the feed temperature increased, the permeation fluxes of both systems were increased. The selectivity of the polysulphone coated membrane was higher than that of the cellulose acetate coated membrane.

*Symposium [J]
16:00-18:30, Jan. 8*

Composition Development and Synthesis of Novel Ceramic Membranes for Oxygen Separation and Cathodic Applications

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Systems within the $(\text{LaSr})\text{FeCoO}_3$ have been developed and characterized as oxide conducting (ionic/electronic) double oxides with several potential electronic applications, including cathodes for Solid Oxide Fuel Cells, and Oxygen Separation Membranes. Oxygen transport occurs through the membrane by solid state diffusion, the imposed oxygen nonstoichiometry, when the membrane is placed in an oxygen activity gradient. The increase in interfacial polarization, due to charge build-up, is circumvented by the inherent partial electronic transport. The composition $(\text{La}_{0.8}\text{Sr}_{0.2})(\text{Fe}_{0.6}\text{Co}_{0.4})\text{O}_3$ (LSFC) has been characterized and identified as a potential candidate for oxygen separation membranes due to its extended range of oxygen deficiency and relative stability at low oxygen activity.

In dense films (2-10 μm) of LSFC have been deposited on dense and on porous ceramic substrates by using a novel liquid precursor/spin coating technique. The thickness of the deposited film depends on the viscosity of the precursor and the opening conditions. The microstructure of the film and its adherence to the substrate depend on the thermal regime.

Synthesis of LSFC films, its composition development and its potential applications as oxygen separation membrane, as a cathode in Solid Oxide Fuel Cells, and in liquefaction of methane, will be presented.

**Biomaterials
and Biotechnology**

Performance of Lactic Acid Based Polymers in Commercial Applications

Patrick R. Gruber

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Commercial applications of poly(lactide) and poly(lactic acid) generally referred to as PLA, requires that PLA be modified in order to meet the demanding requirements of customers. PLA made safety from lactic acid is unacceptable for commercial applications, instead, companies, like Cargill, are developing copolymers and formulated compositions of polymers based on lactic acid in order to target the demanding needs of customers. Factors such as optical composition, melt viscosity, rheology, and shelf stability need to be tailored to meet commercial performance requirements such as processability, regrind, shelf life. Additionally, the physical and materials properties need to be tailored to meet end use product requirements. After formulation, lactic acid polymers have been shown to be useful in a wide variety of applications such as cast film, blown film, injection blow molding, injection molding, fiber, meltblown nonwoven, spunbond nonwoven, thermoforming and other applications. This presentation will contrast PLA and its deficiencies to formulate lactic acid based polymers which are finding success in applicators.

Finally, in meeting the functional requirements for applications, the cost of the polymer is extremely important. In this presentation, the costs to manufacture lactic acid based polymers will be discussed. Economics as well as advantages and disadvantages of competitive processes will be compared.

*Symposium [A]
13:30-15:40, Jan. 5*

Properties-by-Design in Biomedical and Pharmaceutical Materials

Shalaby W. Shalaby

Bioengineering Department

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Poly-Med, Inc., Anderson, South Carolina

Both synthetic and natural polymers can be used more effectively as biomedical and pharmaceutical materials, with strategic modulation of their structure or morphology. Recent developments in exploiting this theme pertain to (a) controlling the compliance, absorption profile, and phase transitions in absorbable polymers; (b) enhancing the radiation stability of absorbable and non-absorbable implants; (c) constructing microporous foam and self-reinforced composites; and (d) controlling the biodegradation and thermal behavior of proteins.

*Symposium [A]
13:30-15:40, Jan. 5*

New Generation of Materials

Ellis Douek, FRCS

Consultant ENT Surgeon, Guy's Hospital, London, U.K.

Eversince a wooden leg was offered to the amputee we have searched for replacement materials when body parts were failed. What has been made available has always been a trade-off. A material showing adequate bio-active with good bonding qualities may have deficiencies in other ways.

Our work has involved the use of bioglass[®] ear. Restoration of hearing often means replacement of the whole ossicles that have been destroyed or damaged. Bioglass[®] lends itself particularly well in this situation as its mass is entirely suitable and the brittleness of glass is of no consequence here.

The use of Bioglass[®] as an ossicular replacement over a long period of time is described here. It has also been used in the form of a cylinder to provide a bone-anchored per-cutaneous connector for the transmission of electrical impulses in cochlear implants.

Another use described in this paper is Bioglass[®] powder as a filler for cavities in the mastoid bone created by surgical intervention.

Advances in Absorbable Sutures

Mark S. Roby

U. S. Surgical Corporation, USA

The Purpose of this presentation is to review the properties of synthetic absorbable sutures and to describe the development of the most recent material, BIOSYNTM sutures.

All the major commercially available synthetic absorbable sutures have a glycolic acid linkage as a major component which is provided by either polyglycolic acid (PGA) or polydioxanone (PDO). Other ring-opening polymers used a copolymer with PGA include polylactic acid (PLA), polutrimethylene carbonate (PTMC) and polycaprolactone (PCL).

The first commercial synthetic absorbable suture, Dexon suture, introduced in 1970, is composed of PGA. Due to a relatively high tensile modulus, PGA fibers must be fabricated into a braid in order to achieve good handling properties. Two other braided synthetic absorbable sutures have since been developed, Vieryl suture and POLYSORBTM suture. These materials are based on PGA/PLA copolymers and have similar properties.

Since the early 1980's, three monofilament synthetic absorbables have been introduced -- Maxon sutures, PDS sutures and Monocryl sutures. These materials are significantly more flexible than PGA.

In vivo strength retention profiles for all of these materials differ widely. While the braids offer 3 weeks of strength, monocrayl suture offer about 2 weeks, Maxon suture about 4-5 weeks and PDS II about 6-8 weeks.

The objective of our development program was to design a monofilament absorbable with an in vivo strength retention profile of a braided absorbable.

A triblock co-polymer comprised of a PDO/PTMC middle block and PGA/PDO end block was found to produce the desired objective. Within this series, it was determined that the middle block-end block composition should be about 40/60 mole% to achieve the required tensile modulus while the PDO/PTMC middle block composition should be about 35/65 mole % to achieve the required in vivo strength retention profile.

*Symposium [B]
16:00-18:00, Jan. 5*

Effect of Composition on the Produced Phases in Dental Amalgam Alloys

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**Faculty of Dentistry, Alexandria University, Alexandria, Egypt*

Six different dental amalgam alloys (A-F) were prepared from their ingredients (silver, tin, copper) by centrifugal casting technique. Ingots were then heat-treated and comminuted. The copper content was varied from 2% to 25% by weight; the first three alloys A, B, and C represent low copper amalgam alloys, while the remaining alloys D, E, and F represent high copper amalgam alloys. The particle size distribution was confined to be in the range of 33μ to 54μ by sieving.

The microstructure of the prepared alloys and their amalgams was investigated using X-ray Diffraction analysis, Scanning Electron Microscopy in conjunction with Energy Dispersive Spectroscopy. It was found that the most predominant phases in low copper alloys were $\gamma(\text{Ag}_3\text{Sn})$, $\epsilon(\text{Cu}_3\text{Sn})$ and $\beta(\text{Ag}_4\text{Sn})$ while in high copper alloys were $\beta(\text{Ag}_4\text{Sn})$, $\epsilon(\text{Cu}_3\text{Sn})$, $\delta(\text{Cu}_{41}\text{Sn}_{11})$ and (Ag)-solid solution.

The phases produced when each alloy powder was mixed with mercury were also studied. It was seen that the γ_2 phase was absent in the high copper amalgams, whereas the $\eta\text{-Cu}_6\text{Sn}_5$ phase was present instead. This was interesting finding is remarkable as the γ_2 phase is the most undesirable phase susceptible to corrosion.

Synthesis of Biodegradable Polyester

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The present paper presents and discusses three different approaches to the synthesis of different classes of biodegradable polyesters.

- The synthesis of polylactides by ring-opening polymerization of cyclic lactides is best performed by the insertion mechanism which evolves reactive covalent bonds, but avoids ionic intermediates. Interesting initiators of the insertion mechanism can be prepared in situ from AlEt_3 , ZnEt_2 , MgBu_2 and various alcohols or phenols. This strategy allows the convenient synthesis of oligo or polylactides showing biologically active end groups.
- The second approach concerns the modification of aliphatic and mainly aromatic polyesters by incorporation of anhydride groups. The rate of hydrolytic degradation can be controlled by the molar fraction of anhydride groups, and thus, optimized for a variety of applications.
- The third approach deals with the synthesis of biodegradable liquid-crystalline (LC) polyesters based on phloretic acid. The properties of these copolyesters can be varied by the structure of the comonomers which are all nontoxic natural products.

*Symposium [C]
10:30-12:30, Jan. 6*

Polymeric Hydrogels as Important Supports to Realize Targetting of Biologically Active Species

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Synthesis of well defined polymeric hydrogels based on vinyl and acrylic monomers is described. Modification of these hydrogels with biologically active species (enzymes, proteins, anticoagulants, cofactors etc...) during the hydrogel formation due to the transformation of these species into macromonomers permits to realize new family of polydrugs, affinic sorbents, biomedical devices, anticoagulant coatings for implants and other materials serving a bridge between synthetic macromolecular chemistry, biochemistry and medicine.

Of special interest is the possibility for immobilization into hydrogels of living microbial and other cells which opens new pathways in biotechnology and creation of biosensors.

Oral insulin delivery to threat diabetes using some polymeric hydrogels is also described.

*Symposium [C]
10:30-12:30, Jan. 6*

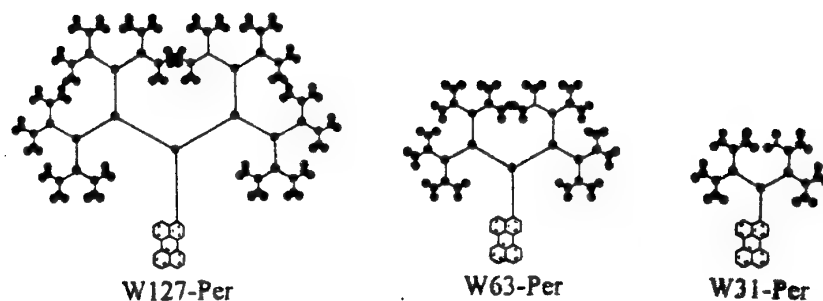
Fiber-Optic Intracellular Bio-Nanosensors and Near-Field Optical and Chemical Imaging

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The smallest optical sensors in use are chemical and biochemical sub-micron "optodes" employing polymeric composites. For instance hydrogel copolymers include fluorescent indicator monomers and serves as hosts for enzymes or doped cyclodextrins. They are prepared in-situ on a pulled fiber-optic nanotip by near-field photopolymerization unaffected by the optical diffraction limit. Alternatively submicron hydrophobic liquid polymer films deposited on such fiber tips entail a carefully designed cocktail of highly selective ionophores fluorescent chromoionophores "inner filter" dyes lipophilic ionic additives PVC plasticizer etc. The submicron probes have outer diameters between 250 and 900 nm. They are mechanically chemically and photochemically robust and have been used routinely for intracellular measurements. They are extremely fast (10 ms response times) and sensitive (zeptomole detection limit). Sensors for pH oxygen glucose calcium sodium potassium chloride etc. have been made with excellent sensitivity for intracellular measurements. Towards the aim of single molecule near-field optical probes and sensors special supermolecule antenna systems have been designed and investigated. Phenyl-acetylene dendrimers up to seven generations serve as such antennas. Highly efficient directed multistep energy transfer has been demonstrated for these molecular energy funnels.



*Symposium [D]
14:00-15:40, Jan. 6*

Cell Behavior on Polymer Surfaces with Different Functional Groups

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Recently a large number of research groups have studied the interaction of biomedical materials with cultured cells because cell-compatible materials are thought to be very important in many biomedical applications. Some of them have focused on the interaction of cultured vascular endothelial cells and biomaterials to improve their blood compatibility when vascular grafts are implanted in the body. Others have studied the interaction of tissue cells with biomaterials to improve their tissue compatibility when the materials are implanted as substitutes for organs, skin, bone, etc.

It is recognized that the behavior of the adhesion and proliferation of different types of cells on polymeric materials depend on the surface characteristics such as wettability (hydrophilicity/hydrophobicity or surface free energy), chemistry, charge, roughness, and rigidity. A large number of research groups including ours¹⁻³ have studied the interactions of different types of cultured cells with various polymers with different wettabilities to correlate the relationship between surface wettability and blood- or tissue-compatibility.

In this study, functional group gradient surfaces where the surface density of grafted functional groups changes gradually along the sample length were prepared on low density polyethylene (PE) sheets by corona discharge treatment with gradually increasing power and graft copolymerization of acrylic acid (AA), sodium-p-styrene sulfonate (NaSS), and N,N- dimethyl aminopropyl acrylamide (DMPAA). AA and NaSS are negatively chargeable and DMPAA is positively chargeable in phosphate buffered saline or cell culture medium at pH 7.3-7.4. The functional group gradient surfaces were characterized by the measurement of water contact angle, Fourier transform infrared spectroscopy in the attenuated total reflectance mode, and electron spectroscopy for chemical analysis. All these measurements indicated that the functional groups were grafted on the PE surfaces with gradually increasing density of them.

The interaction of Chinese hamster ovary cells with the functional group gradient surfaces along the sample length was investigated. The cells adhered and grown on the surfaces were counted and observed by scanning electron microscopy. It was observed that the cells were adhered and grown more onto the positions with moderate density of the functional groups. This may be related to the hydrophilicity of the surface. The DMAPAA-grafted surface showed large amount of cell attachment probably due to the positive charge character, while the AA-glazed surface which is charged negatively showed poor cell attachment, as expected. The NaSS-grafted surface which is also charged negatively showed large amount of cell attachment. This may be closely associated with the existence of an aromatic ring close to the ionizable group in NaSS. It seems that surface functional groups and their charge character as well as wettability play important roles for cell adhesion, spreading, and growth.

*Symposium [D]
14:00-15:40, Jan. 6*

Bioartificial Polymeric Materials

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In spite of the great improvement of the biological performances of many purposely designed new polymeric biomaterials, their interactions with living tissue components remains the major problem to be solved.

It could be interesting to smooth away the interactions between the synthetic and the biological systems by preparing materials in which changes at molecular level, due to synthetic-biological polymer interactions, have been already accomplished before getting in touch with the living tissues. Such a material, with already established molecular interactions, should behave macroscopically better than a fully synthetic material as far as the biological response of the host is concerned.

In addition, the study of molecular level interactions would allow one both to select at a very early stage those synthetic polymers that will be potentially used in making new polymeric biomaterials, and to get information about the biocompatibility characteristics of the polymers. The design of new materials based on blends of biological and synthetic polymers started in our laboratories, the final purpose being the production of new processable polymeric materials that possess both good mechanical properties and good biocompatibility characteristics. We also proposed that this class of materials be called "bioartificial polymeric materials".

Bioartificial materials have been prepared, in different forms (films, hydrogels, sponges), by mixing natural polymers as collagen, hyaluronic acid or its ester derivatives, starch etc., with various commercial synthetic polymers as poly(vinyl alcohol), polyurethanes, poly(acrylamide), etc.

Recently, new bioartificial materials have been obtained starting from blends, of gelatin and poly(vinyl alcohol) (PVA) and of dextran and poly(acrylic acid) (PAA).

The materials have been prepared in form of films, using the solution casting method, and characterized by different techniques in order to evaluate their mechanical, thermal and morphological behavior. The results obtained, are illustrated and the different properties showed by the two systems are evidenced.

Biodegradation and Medical Application of Chitosan Hard Capsule

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Chitosan is an amino polysaccharide derived from chitin which is the major constituent of exoskeleton of crustaceous animals. This biopolymer is an interesting material in the development of new application to medical use because it is nontoxic, highly biocompatible and biodegradable.

In this study, chitosan hard capsules containing barium sulfate and coated with enteric coating agent were orally administered to six beagle dogs and X-ray photographs were taken. All capsules degraded only in colon. In addition, chitosan hard capsules containing salicylamide (SA) and gelatin hard capsules containing riboflavin (VB2) as control were simultaneously administered orally to 31 year-old adult males. The time of maximum urinary rate (T_{max}) of SA and VB2 was 13 and 3 hours, respectively. In another examination, chitosan hard capsules containing 20 IU insulin and Na-glycholate were administrated to wister rats. The chitosan hard capsules containing insulin and Na-glycholate were effective for reducing the plasma glucose levels compared with gelatin hard capsules containing same components.

These results suggest that the chitosan hard capsules can be used for a new colon-specific drug delivery system.

*Symposium [F]
10:30-12:00, Jan. 7*

A New Class of Swelling Type Materials for Bioimplant Applications

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A new class of swelling type composite material system is being developed for bone implant applications. This material system is expected to provide better short and long term fixation characteristics when compared against traditional cemented and non-cemented techniques. Controlled swelling of the implant produces a compressive pressure on the entire bone-implant interface as well as a smooth load transfer from the implant to the bone. This compressive pressure will enhance frictional resistance to sliding between bone and implant, and thereby provides good fixation characteristics in the short term. The compressive interfacial pressure also introduces a tensile hoop stress in the bone, which will induce bone densification and bony in growth at the bone-implant interface, making the bone-implant system act as an integral unit in the long term.

The composite material system developed in this study comprises of a copolymer of methylmethacrylate (MMA) and acrylic acid (AA), which may be optionally reinforced with a three-dimensional network of graphite fibers for high strength, high toughness, applications. The details of processing these materials (both the copolymer as well as its composite) along with the resulting microstructures, swelling characteristics, and mechanical properties are presented in this paper. It was observed that by suitably controlling the relative amounts of MMA and AA, and through introduction of cross-linking, the swelling and the degradation in mechanical properties accompanied with swelling can be suitably controlled in this class of materials. Fixation strengths of these materials in bone have been evaluated and found to be superior to many of the currently used implants.

*Symposium [F]
10:30-12:00, Jan. 7*

Microbial Synthesis, Physical Properties and Biodegradability of Poly(hydroxyalkanoates)

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Aeromonas caviae produced a random copolymer of 3-hydroxybutyric acid(3HB) and 3-hydroxyhexanoic acid (3HH) under aerobic conditions when sodium salts of alkanolic acids of even carbon numbers ranging from C₁₂ to C₁₈ or olive oil were fed as the sole carbon source. The weight-average molecular weights of P(3HB-co-3HH) were in the range (2-11) x 10⁴Da. The structure and physical properties of P(3HB-co-3HH) with compositions of 5-25 mol% 3HH were characterized by ¹H and ¹³C NMR spectroscopy, X-ray diffraction, differential scanning calorimetry, mechanical tensile measurement, and optical microscopy. The degree of X-ray crystallinity of solvent-cast P(3HB-co-3HH)films decreased from 60 to 18% as the 3HH fraction was increased from 0 to 25 mol%, suggesting that 3H units are excluded from the P(3HB) crystalline phase. The rates of enzymatic erosion increased markedly with an increase in the 3HH fraction to reach a maximum value at 15 mol% 3HH, followed by a decrease in the erosion rate.

A partial fragment of *A. caviae* PHA synthase structural gene(*phaC_{Ac}*) was amplified by PCR with primers designed from the highly conserved regions among known PHA synthases. A cosmid library of *A. caviae* gDNA was screened by colony hybridization with the PCR-amplified fragment as a probe, and several positive clones were isolated. The *phaC_{Ac}* was mapped on a 11-kbp *Sal*I fragment and a nucleotide sequence of 5.0-kbp *EcoRV*-*EcoRI* fragment was determined. The *phaC_{Ac}*(1782bp) encoded a protein composed of 594 amino acids with a molecular weight of 66,334 Da.

Symposium [G]
16:00-18:00, Jan.7

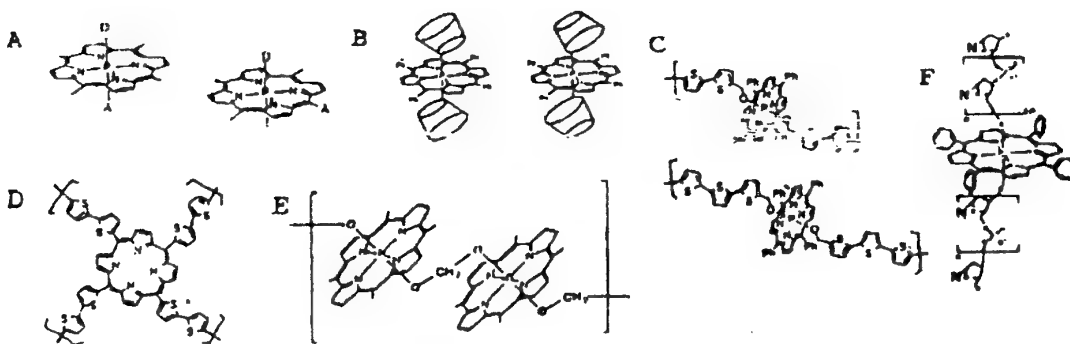
Molecular Engineering of Porphyrin

T. Shimidzu

Kansai Research Institute, Kyoto, Japan

Molecular Engineering, tailor-made systematization of functional molecule is an important research subject for functional molecular materials. In principle, some of the multiporphyrin systems are considered to be converted into the elements of molecular photo-electronic devices. Especially, their systematization with appropriate electron mediators into large polymeric systems is one of the feasible approaches to the molecular systems based on the electron transfer. Phosphorous (V) porphyrin has a unique structure. 3 centers 4 electrons, covalent bond in axial direction, and many interesting derivatives were synthesized.

They are: a donor-sensitizer-acceptor triad molecule (A) for an efficient photo-induced electron transfer a cyclodextrin substituted P(V) porphyrin (R) for efficient photo-chemical traducing catalysis, 1-D and 2-D porphyrin arrays connected with conjugation molecular wire (C,D) for molecular electronics, 1-D porphyrin arrays connected with insulating molecular wire (E) for molecular photonics, and an oligonucleotide shackled with porphyrin (F) for artificial photo active restrictive enzyme. They all showed their specific attractive functions as the authors had expected.



*Symposium [G]
16:00-18:00, Jan. 7*

Production of Poly(3-Hydroxybutyrate) by Recombinant Bacteria

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Polyhydroxyalkanoates (PHAs) are carbon and energy reserve material produced by numerous microorganisms under unfavorable growth conditions. PHAs have been drawing much attention as candidates for biodegradable plastic and elastomer material. Among these, poly (3-hydroxybutyrate) [PHB] is most frequently found membrane of the PHAs, and has material properties similar to those of polypropylene. Recombinant *Escherichia coli* strains harboring a high copy number plasmid pSYL105, which contains the *Alcaligenes eutrophus* PHA biosynthesis genes were constructed, and were used for the production of PHB to a high concentration. Considerable filamentation occurred during the synthesis of PHB could be suppressed by the introduction of *ftsZ* gene in a multi-copy-number plasmid. This filamentation-suppress strain of recombinant *E.coli* was shown to synthesize polymer more efficiently in a defined medium compared with the strain that undergoes filamentation. Fed-batch culture was carried out to obtain more than 70 g/L of PHB in a defined medium with high productivity. Detailed results on the production of PHB by various recombinant *E.coli* strains will be presented.

Symposium [G]
17:00-18:00, Jan. 7

Crystallite Size and Strain Developed in Laser Irradiated Dentine and Enamel

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Human dentine and enamel were prepared, powdered and irradiated, in vitro by 3 types of low energy density laseres. Pulsed lasers on CO₂, Nd:Yag and Ar with separate energies of 15 and 30 j/cm² were used. The induced effects on crystallite size and strain were calculated from the low speed scan X-ray diffraction analysis using the line broadening method. The data were complemented by scanning electron microscopy before and after laser irradiation and related to their dissolution behavior in lactate buffer media.

Results showed that CO₂ is the most effective one. The behavior of crystallite size of dentine showed reserve change to strain values at both energies. Nd:Yag laser irradiation gave similar values to the unirradiated crystallite sizes and strain parameters. On the other hand, the enamel showed parallel decrease in the value of both parameters upon laser irradiation with CO₂ the most effective in reducing the strain to decrease its half and third values with 15 and 30 j/cm² respectively. Higher resistance to dissolution especially with CO₂ laseing was attribute to its higher absorption compared to others. The difference in response of both tissues was attributed to their original structures and chemical nature. The biological implication of material disorder upon laser treatment was verified.

*Symposium [G]
16:00-18:00, Jan. 7*

Preparation and Application of Cationic Polysaccharides in Cosmetic Formulations

**William H. Daly, Melissa Manuszak-Guerrini,
Doris Culberson and Javier Maccossay**
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Cationically charged water soluble saccharides exhibit properties particularly suited for cosmetic formulations. A naturally occurring aminopolysaccharide, chitosan has been used in various hair products, but its use is limited due to insolubility in the common solvent systems employed, i.e. water, alcohols and neutral to alkaline detergent solutions. Procedures for hydroxypropylating chitosan to yield derivatives soluble in neutral aqueous mixtures facilitate applications in more diverse formulations. Treatment of chitosan with N-(3-chloro-2-hydroxypropyl)trimethylammonium chloride (Quat 188) affords a water soluble quaternary derivative with excellent solution properties. Each of these chitosan derivatives could be employed in cosmetic formulations.

The synthesis of water and alcohol soluble aminoalkyl carbamoyl cellulose derivatives by aminolysis of either cyanoethylated cellulose or amidation of methyl carboxymethyl cellulose has been reported]. Converting commercially available carboxymethyl cellulose to a methyl ester produces a derivative more receptive to amidation. When the methyl carboxyl-methyl cellulose is treated with an excess of diamines, $RN(CH_2)XNR'R''$ where $R, R', R'' = , CH_3$, water soluble aminoamide cellulose derivatives are obtained. Further reaction of tertiary aminoamide cellulose derivatives with Quat 188 produces bis-quaternary salts with unique properties.

Application of these polymers in cosmetic formulations requires an understanding of their solution properties, particularly their interaction with surfactants. The quaternary polysaccharides described above are used to determine the interaction between relatively rigid quaternary polymers and mixed micelles of sodium dodecyl sulfate (SDS) and Octoxynol (Triton X-10). The results of precipitation, fluorescence, and light scattering studies comparing the behavior of these polymers with a quaternary polymer currently used in the industry, Polyquaternium 10, will be reported. The precipitation studies reveal that the concentration of anionic surfactant required to induce precipitation of the quaternary polysaccharides from dilute solution is lower than that used for Polyquaternium 10.

The fluorescence studies the formation of polymer- mixed micelle complexes. Variable temperature light scattering studies show that solutions of the new quaternary polysaccharides-SDS-Octynol complexes are more thermally stable than those derived from Polyuaternium 10. Surface tension studies of the polyquat-surfactant complexes demonstrates a synergistic lowering of the surface tension at very low concentrations of SDS which implies the formation of a highly surface active polymer-surfactant complex. A corresponding increase in the foaming power of the solutions is also observed.

*Symposium [K]
10:30-12:10, Jan 9*

Synthesis and Antitumor Activities of Polymers Containing Amino Acids or 5-Fluorouracil

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The polymeric drugs can be expected to have some advantages such as higher specificity of actions, longer duration of actions and lower toxic side effects compared with low molecular weight drugs.

Butler et al synthesized the copolymer (DIVEMA) of divinyl ether with maleic anhydride, and it has been extensively studied for its structure and broad biological activities. We also have been reported on the syntheses and biological activities of polymeric antitumor agents.

The aim of this work is to synthesize new biologically active polymers containing amino acids or 5-fluorouracil (5-U). In this study, the new monomers, cis-5-nobornene-endo-2,3-dicarbonyl derivatives (NDCA) having amino acids moiety were synthesized by the reaction of cis-5-nobornene-endo-2,3-dicarboxylic anhydride (NDA) and amino acids, and NDCA containing 5-FU (NDCA-FU) was prepared from NDCA and 5-FU.

The homopolymers and copolymers with acrylic acid (AA) or vinyl acetate (VAc) were synthesized by photopolymerizations in photochemical chamber using 313 nm u.v lamps at 25 C for 40 h. The structures of synthesized monomers and polymers were identified by FTIR and ¹H-NMR spectrophotometer. The number and weight average molecular weights were determined by GPC.

The *in vitro* antitumor activities of prepared polymers were evaluated against mouse mammary (FM3A), mouse leukemia (P388), and human histiocytic lymphoma (U937) as cancer cell lines. The *in vivo* antitumor activities of synthesized samples against Balb/C mice bearing sarcoma 180 tumor cell line were also evaluated.

Symposium [K]
10:30-12:10, Jan.9

Drug Release from Some Hydrophilic Polymer Matrices

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Polymers are widely used in medicine as materials for sutures, artificial organs, orthopedic implants, and devices for the controlled release drugs. Controlled drug delivery provides two important potential applications for the use of polymers in the effective management of medical drugs in the body. The first of these is controlled release where a steady therapeutic concentration of the drug is maintained. The other application is site-directed drug delivery whereby a polymer serves as a carrier to bring a drug to a specific site in the body. The rate of drug release can be controlled by diffusion, reaction, or solvent. In diffusion control, the driving force for diffusion is the concentration gradient across the delivery device, which can be a reservoir or matrix system. In this work, release of Metoclopramide Hydrochloride and Cisapride from crosslinked Sodium Alginate matrix was studied. It was shown that the release rate is influenced by the crosslinking technique of the matrix film, crosslinker type, drug physico-chemical properties, activity of the release medium, concentration and the loaded quantity of the drug in the matrix. The crosslinking process of the matrix film was shown to be an interfacial phenomenon and the nature of crosslinking depends on the crosslinker type and concentration. This work also showed that crosslinked alginate in a matrix form has limitation in practical use due to the effect of acidic medium on the crosslinking and rate of drug release.

*Symposium [K]
10:30-12:10, Jan.9*

**Degradation
Stabilization
and Recycling**

Wavelength Sensitivity in the Photodegradation of Polymers

Anthony L. Andradý

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Wavelength sensitivity of light-induced damage to polymers can be studied under monochromatic or polychromatic exposure conditions. The former simpler exposure experiments yield action spectra for the various degradation processes. These show a logarithmic dependence of the efficiency of the yellowing discoloration on the wavelength of irradiation. Using a xenon write light source and a series of cut-on filters activation spectra indicating the wavelength sensitivity of selected photoprocesses to various wave bands of the source spectrum can be determined. Activation spectra are source-specific but take into account synergistic (or the opposite) effects of two or more wavelengths in the source spectrum. The theoretical basis for obtaining such spectra and the validity of interconversion of action and activation spectral data will be discussed in this presentation. In generating wavelength sensitivity data as well as in interconverting spectral data certain assumptions on the intensity dependence of photodamage, reciprocity, and the time dependence of the degradation process are made. In most instances these assumptions have not been tested with the polymer systems of interest.

Photodegradation data of poly (vinyl chloride), polycarbonate, polystyrene, and polyolefins will be used to illustrate these phenomena. Specific photodamage processes of interest include change in Yellowness index of the polymers and changes in average tensile extensibility of the polymers. Activation spectra discussed will be based on filtered xenon sources that simulate terrestrial solar radiation.

*Symposium [E]
16:00-17:55, Jan. 6*

Polyolefin Stabilization, from Single Stabilizers to Complex Systems

F. Gugumus

Ciba-Geigy AG, Basel, Switzerland

The high efficiency of Hindered Amine Stabilizers (HALS) in polyolefins has been well documented in the past 20 years. It is less well-known that this performance can still be improved significantly by an adequate choice of the stabilizers. This additional improvement is achieved by combination of different light stabilizers or of light stabilizers with costabilizers. The optimum combination depends on the particular polyolefin and the particular application envisaged. With respect to the application, sample thickness and relevant test criterion are determining to a large extent the choice of the stabilizer system.

It is found that, in thin cross-sections such as PP tapes, combinations of HALS with light stabilizers of a different type do not usually yield synergistic effects. However, combinations of two light stabilizers of the HALS-type can yield pronounced synergistic effects. The performance of several of these combinations is illustrated with data generated in PP tapes, PP multifilaments and PE-LD films. Depending on the application, combinations of low molecular mass HALS with high molecular mass HALS or polymeric HALS, or combinations of two high molecular mass HALS show the best effects. Combinations of HALS also yield synergistic effects in thick cross-sections, the domain of preferred use of UV absorbers. The efficiency of HALS combinations in polyolefin thick sections is illustrated with examples involving PP as well as PE.

There is another aspect of polymer protection by HALS which is much less known than their contribution to UV stability. In fact, HALS are protecting polymers in the solid state from thermal oxidation as well as from photooxidation. The contribution of HALS to long-term thermal stability of polyolefins is discussed in detail.

*Symposium [E]
16:00-17:55, Jan. 6*

Photooxidation of Unstabilized and Stabilized Polyphasic Ethylene Propylene Polymers

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The oxidation photoproducts resulting from the UV irradiation of a polyphasic polymer PE/EPR in conditions of artificial accelerated exposure have been identified by different analytical methods. These methods included FTIR analysis of the polymeric matrix coupled with derivatization reactions. The identification of low the molecular weight photoproducts was carried out by chromatography and mass spectroscopy analysis. It was shown that the oxidation photoproducts were those of the polypropylene phase, which is by far the most oxidizable. Moreover, our results showed unambiguously that the formation of the photoproducts was resulting from an oxidation of the tertiary carbon atom, which is consistent with expected reactivities.

The evolutions of the FTIR spectra of 100 μm films exposed to artificial accelerated photoageing were compared to those of thick plaques (3mm) submitted to natural outdoors weathering and analyzed by photoacoustic (PAS)-FTIR spectroscopy. This comparison showed the relevancy of the phenomena observed in the conditions of artificial aging and permitting the determination of an acceleration factor for the main oxidation route.

The same experiments were reported with samples stabilized with a low molecular weight hindered amine light stabilizer and a high molecular weight stabilizer. Depth profiling was achieved by mass-FTIR spectroscopy. This technique permits the analysis of the distribution of the products in the oxidized polymeric matrix. It was shown that the behavior of the stabilized samples was largely depending on the migrating possibilities of the additives, specially as far as thick samples were concerned. Analysis of the outer surface layers of samples stabilized with the low molecular weight additive indicated that a grafting reaction occurred by interaction of nitroxyl radicals and oxidized macroradicals.

*Symposium [E]
16:00-17:55, Jan. 6*

Corrosion Protection by Tin Deposited by Different PVD and PACVD Techniques on Steel

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Coating with TiN improves the surface properties, such as hardness, wear and corrosion resistance of steel. Although the various PVD (physical vapor deposition) and CVD (chemical vapor deposition) methods have been developed, the effort still has been made to raise the efficiency of the methods and to improve the quality of coatings. The main goals are:

1. The decrease of deposition temperature in order not to change the substrate structure and properties, and
2. The production of coating of similar thickness all over the covered surface.

By PVD technique, TiN may be deposited at about 400°C, whereas by CVD method, the temperature of 800-900°C is necessary to obtain a coating of high quality. However, CVD technique is relatively cheap and provides the uniform coating of parts of complicated shape. Application of plasma during chemical deposition (PACVD) allows to decrease the deposition temperature. The placing of coated parts in different section of reactor provides further possibility to modify the coating structure and thus their corrosion protection.

The corrosion resistance of TiN formed by means of cathode arc plasma deposition (CAPD), hot hollow cathode deposition (HHCD) was compared with TiN deposited by PACVD method at different parameters. The corrosion resistance of coatings was evaluated by means of electrochemical measurements in H_3PO_4 and NaOH solutions.

Although the PACVD layers (formed using $TiCl_4$) contain some amount of Cl, by choosing the deposition parameters, the TiN layers of higher corrosion protection than those formed at PVP processes could be obtained. High corrosion protection of PACVD layers is accounted by their good adhesion. PVD TiN coatings exhibited pores and flaws decreasing corrosion protection.

Polymer Surface Chemistry Induced by Environmental and Biological Milieus - Degradation vs. Reversible Reorganization

Joseph A. Gardella, Jr., Hengzhong Zhuang*,

Professor of Chemistry and Director, Materials Research Instrument Facility,

**Marc Patterson, John Strong, and Jiaying Chen, Department of Chemistry
SUNY Buffalo, Buffalo, NY, USA 14260-3000*

Recent research into the near surface microphase separation of multicomponent copolymers has elucidated the surface structure and composition of a wide variety of technologically important materials. Many multicomponent materials are used as protective coatings, and are exposed to complex environmental and biological challenges. Our research program has investigated a wide variety of copolymers employing fluorocarbon or siloxane surface active components to provide low surface energy materials for biomaterials and minimal fouling marine coatings. The goal of our work is to quantify the surface structure and composition of the near surface region, including a description of the microphase separated domain structure which evolves near the interface of a prepared film.

Recently we have developed approaches to describe these surfaces after exposures to various biological and environmental challenges. These challenges are manifested in two behaviors of direct interest to polymer chemists; degradation at the surface and reorganization in the near surface region. Both reactive effects yield a surface with very different properties.

The present paper will focus on two areas. Surface reaction kinetics are an important result of the designed **in-vitro** degradation of biodegradable polymers, synthesized with various hydrophobic and hydrophilic components. The determination of reaction degradation kinetics at short times can allow for an understanding of the use of such materials for drug delivery.

A second area of interest is the development of the non-fouling marine coating based on polyurethane and poly siloxane chemistries. Siloxane surface structures provide good non-fouling characteristics initially, but do not appear stable under water. A detailed examination of the reorganization of siloxane-polyurea urethane polymers induced by simulated marine exposure shows the extent and speed of surface reorganization.

*Symposium [H]
10:30-12:30, Jan.8*

Prediction of Polymer Lifetime in Harsh Climate

Halim Hamid

*King Fahd University of Petroleum and Minerals,
Dhahran, Saudi Arabia*

The weathering of polymers is dependent upon all parameters of environment. The weather varies from place to place and from time to time particularly in the Gulf region. The need of lifetime prediction of polymers for a certain location is of high technical and economic importance to the plastics producers, processors, and the end-product consumers.

The natural exposure trials on the linear low density polyethylene (LLDPE) samples were carried out at Dhahran, Saudi Arabia. The artificial weathering data have been obtained by exposing the samples in ATLAS weatherometer. Changes in the properties of exposed LLDPE samples were monitored using Fourier Transform Infrared (FTIR) spectroscopy, differential scanning calorimetry (DSC) and mechanical testing system.

A mathematical approach for describing the weather-induced degradation of linear low density polyethylene (LLDPE) has been adopted to understand the degradation behavior of polyethylene. A correlation between natural and artificial weathering has been developed for lifetime prediction of polymers exposed for a shorter period.

*Symposium [H]
10:30-12:30, Jan.8*

New Trends in The Stabilization of Poly(vinyl chloride) Against Thermal Oxidative Degradation

Ahmady A. Yassin

*Department of Chemistry, Faculty of Science,
University of Cairo, Egypt.*

Poly(vinyl chloride), PVC, suffers from poor heat stability, its degradation occurs by an auto-catalytic dehydrochlorination reaction induced at the labile sites in the polymer chains. A wide variety of metallic soap stabilizers are used industrially to improve the thermal stability of the polymer. However, irrespective of their efficiency, they suffer from the deleterious effects of the by-products, mostly metal chlorides, accumulated during the reaction of these stabilizers with the polymeric chains.

Recent investigations have shown that certain organic materials are able to be as effective stabilizers for PVC as are the metallic stabilizers. Moreover, these organic stabilizers have been found to be able to form metal complexes with the metal chlorides formed as by-products from conventional stabilizers. These complexes are of comparable stabilizing efficiency as are the parent stabilizers. Examples for these organic stabilizers are given together with the mechanisms of their action.

*Symposium [H]
10:30-12:30, Jan. 8*

**Preparation and Evaluation of some
New Corrosion Inhibitors for Varnishes
Part 1: Epoxidized Fatty Materials Condensed with
Sulfur Containing Compounds**

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*Polymer and Pigments Department
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Soya bean oil was epoxidized In-situ under well established conditions. The produced epoxidized soya bean oil was subjected to react with a sulfur containing compound of the general formula (5-phenyl-1,3,4-oxadiazole-2-(3H)- thiones) and four of its derivatives (p-chloro-, p-iodo-, p-methyl- and p-methoxy-) in sealed ampoules under inert atmosphere at 130°C. The produced adducts were added to three varnishes based on chlorinated rubber, alkyd resin and a vinyl chloride copolymer, in different concentrations, to evaluate them as corrosion inhibitors for steel surfaces. It was found that these adducts can act as good corrosion inhibitors and their efficiencies depend principally on the substituted functional group.

*Symposium [H]
10:30-12:30, Jan.8*

Wavelength Sensitivity in the Photodegradation of Polymethylmethacrylate: Accelerated Degradation and Gel Formation

Ayako Torikai

*Department of Applied Chemistry, School of Engineering,
Nagoya University, Japan*

We have been investigating the wavelength effect on polymer degradation using Okazaki Large Spectrograph (OLS) which was build at the National Institute for Basic Biology in Okazaki, Japan. These studies contribute to identify the spectral region which causes damage on polymer materials. Polymethylmethacrylate (PMMA) is widely used for its clarity, stiffness and other excellent characteristics. This presentation will report the wavelength sensitivity in photodegradation of PMMA and the effect of benzophenone (BP) or β -carotene on PMMA degradation.

PMMA and additive containing PMMA films were cast from acetone solutions. Films were irradiated with monochromatic radiation from OLS. After the irradiation, the degradation was estimated by UV-Visible, FTIR spectra (changes of chemical structure) and GPC measurements (main-chain scission). In the case of insoluble fraction was formed, gel measurement was also carried out.

PMMA is known to be a degradation type polymer to photo-irradiation. On irradiating PMMA at ambient temperature in the presence of oxygen, the threshold wavelength of main-chain scission in PMMA was found to be below 320 nm in our precious studies. BP and β -carotene have one of their absorption maxima at around 340 nm and 450 nm, respectively. So, in photoirradiation of PMMA films containing BP or β -carotene with radiation of longer wavelength than 320 nm, we can expect photosensitized main-chain scission and other types of photo-induced reactions than main-chain scission.

PMMA-BP system: On irradiation of PMMA films containing BP in air, the sensitized main-chain scission and photocrosslinking took place simultaneously. These reactions are dependent on irradiation wavelength. The threshold wavelength for both reactions was found to be around 380nm. The number of main-chain scission and amount of gel increased with the increase of BP concentration in PMMA. Photosensitized main-chain scission favors with the irradiation of radiation at 280 nm and photocrosslinking takes place efficiently with the exposure of 340 nm radiation. A probable scheme for photosensitized reaction will be presented.

PMMA- β -carotene system: Photosensitized main-chain scission by the additive was also confirmed in this case. The threshold wavelength of main-chain scission shifted to around 400 nm. This reaction is also concentration dependent. No gel formation was observed.

Water-Soluble Polymers and the Need for Biodegradability

Graham Swift

Rohm and Haas Company, Philadelphia, PA, USA

Water-soluble polymers are widely used in a myriad of commercial applications. These include consumer products and biomedical applications. Both potentially present similar problems with regard to environmental exposures. Polymers designed for consumer markets are frequently disposed of into the aqueous environment after use where they may biodegrade, remain as recalcitrant xenobiotics, or cause an environmental imbalance, which may be addressed. Biomaterials, on the other hand, are developed as drugs carriers, tissue replacements, and organ replacements, for examples. However, the in vivo environmental response to these polymers must also be understood and harmful side-effects avoided. In both cases thorough testing and precise analysis of degradation intermediates is required to establish that a particular polymer is or not acceptable for environmental exposure.

This paper, while recognizing the above similarities of biomaterials and commercial consumer polymers, will focus on the latter. The similarities, though, should be understood by researchers in both fields since there is synergism to be gained by understanding the common problems and solutions.

Interest in biodegradable polymers, particularly oligomers, is several decades old. The detergent industry long ago recognized the non-biodegradability of branched alkyl chain surfactants and substituted them with linear analogues. Similarly, widely used poly (alkylene glycol)s have been thoroughly evaluated for biodegradability. However few water-soluble polymers have been developed that meet all the stringent requirements to claim complete biodegradability.

In this paper, I will discuss the evolution and synthesis of biodegradable water-soluble polymers and the emergence of meaningful definitions test protocols to lend credence to the claim of biodegradation. I will close with a projection of what the future holds for this fields.

*Symposium [I]
13:45-15:45, Jan. 8*

Degradation Studies of Activated Polyethylene in Polyethylene-Starch Polyblends

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Polyethylene is a polymer very much used as packaging material, but it is inert polymer. Much effort has been made to modify polyethylene, e.g. by blending with starch to obtain a photo-and biodegradable blend.

Previous research in this field revealed that by using several types of surfactants as additives and applying several types of micro-organisms biodegraded primarily the starch and only a small part of the polyethylene namely the end groups.

In this work, activation of the polyethylene prior to polyblending has been carried out, i.e. (i) by UV irradiation, (ii) by activation using strong acids; and (iii) by addition of a Ce^{4+} salt as photosensitizer.

Ultraviolet irradiation was carried out at $\lambda = 254$ nm with a variation of exposure time of 10, 15 and 20 days. After that a polyblend was made with a composition of PE : starch = 2:1, and a cationic surfactant as compatibilizer. Characterization was done by FTIR analysis, intrinsic viscosity after hydrolysis of the polyblend, DSC, swelling and SEM. The results showed an increase in molecular weight after 10 days of exposure due to crosslinking and a decrease after 15 days' exposure due to chain scission. Crosslinking in the polyblend makes biodegradation difficult.

Activation of polyethylene by a mixture of chromic acid and nitric acid influences the photodegradation of the polyblend, which is shown by a decrease in melting point and molecular weight.

Activation using a Ce^{4+} salt as photo sensitizer seems to be more effective because this salt is mixed with the polymer, while using acids the polymer must be soaked in the acid mixture. From the FTIR diagram a carbonyl group appears after 15 days' exposure towards UV light.

So activation of polyethylene seems to be able to promote photo- and biodegradation of polyethylene-starch polyblends.

*Symposium [II]
13:45-15:45, Jan.8*

Corporate Management in The Age of Global Environment Awareness -A Case Study of PET Bottle Recycling Issues in Japan

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The global environmental issues are key factors for the business development toward the year 2000. From the industrial ecology perspectives, the management strategy of industrial ecology should be implemented as an essential tool in the decision-making process of top management policy in chemical industry in order to respond to the global environmental challenges. The focus of effort of recovery and recycling of both industrial material of pre-consumer solid waste and post-consumer solid waste become more important in recent years. As a case study, the current problem of recycling of PET bottle business in Japan is reviewed and analyzed in this paper.

To cope with the global issues of alliances for environmental sustainability, the social systems of ecology in our local communities such as recycling post-consumer solid wastes should be established. In order to maximize productivity of global environment, the closed system of recycling materials should be re-evaluated and established as new social and industrial systems. Global environmental productivity is defined as the ratio of total output to total consumption of materials and energy. concepts such as "Seaborg's Closed System" and "Inverse Factories" should be considered if environmental productivity is to be maximized.

The R&D expenditure of both environment and energy saving in Japan reached to 200- 350 billion yen for each project in 1995. It is needless to say that the global environment must be investigated under collaboration of geographically distributed nations, and it should be stressed more that this subject must be dealt with people of different cultural and/or societal background. To encourage large market for recovered material, a growing "buy recycled" movement has emerged. Government and industry have announced purchasing preference for products which contain post-consumer recycled materials where they are available at required price, performance and quality. The environmental soundness of products can be enhanced through "Total Quality Management" (TQM).

*Symposium [J]
16:00-17:55, Jan.8*

Polyamide 66 and 6 Recycling : Technical and Economic Aspects

H.Peter Kasserra

Dupont Nylon, Dupont Canada Inc., Kingston, Ontario, Canada

Resource conservation is a major driving force that encourages recyclability or recycle content of consumer products thereby reducing the amount of waste going to landfill as well as creating new markets for recycle products. DuPont has a strong environmental commitment and as a major producer of polyamide-based polymers, fibres and engineering resins has undertaken the development of a practical system for the recovery of polyamide 66 and 6 from post-consumer products. Our initial attention has been focused on recycling carpets which represent a significant landfill component in North America.

This talk will address the steps that DuPont has taken in creating a closed-loop process and infrastructure for the recycling of carpets. Emphasis will be placed on an integrated business concept, economic drivers and associated technologies. These include carpet collection, sorting and refining, the use of recovered nylon in engineering resins products as well as our proprietary chemical process for recovering pure nylon 66 and nylon 6 monomers. The latter process has been successfully demonstrated through all stages including manufacture of 25% recycle content carpet samples starting with mixed post-consumer nylon 66 and 6 carpets. The application of recovered nylon 66 in engineering resins became commercial last year.

*Symposium [J]
16:00-17:55, Jan.8*

Recovery of Sizes Based on Polyvinyl- Alcohol-Starch Graft Copolymer Using The Ultrafiltration Technique

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The recoverability of polyvinyl alcohol-starch graft copolymer (sizing agent) as well as N-Methylolcarbamoyl ethylated polyvinyl alcohol (reactive size) was studied. The recycling was performed by making use of ultrafiltration technique (UF). Sizing samples in the form of films were subjected to aqueous and/or thermal conditions similar to conventional sizing and desizing conditions, after which the UF technique was applied to reconcentrate the aqueous desized sample solution (from ca. 2 wt.-% to 10 wt.-%). This process was carried out 5 times on each substrate (size). After every cycle the changes in viscosity, solubility, hardness and molecular weight distribution of each substrate were determined. This was done to discover changes in size properties brought about by the high temperature used during sizing and desizing in addition to mechanical erosion caused by the ultrafiltration process. The UF data obtained for every UF cycle [i.e., average permeate flux ($V, L/h.m^2$) and total loss in sizing agent ($L_s, \%$) were also calculated in order to determine the performance of recycling of these substrates by ultrafiltration.

It has been found that PVA-starch graft copolymer synthesized from low molecular weight PVA (PVA-DT) and highly hydrolyzed starch (H_2 -St) fulfills the requirement as a recyclable sizing agent since it possesses higher stability and higher ability to recycling with minimum erosion.

*Symposium [J]
16:00-17:55, Jan.8*

Recycling Plastics in Fiber Reinforced Polymer for Packaging of low Level Radioactive Wastes (LLW).

M.E. Tawfik and N. Ikladios,
*Polymers and Pigments Department,
National Research Centre, Dokki, Cairo, Egypt.*

Recycled poly (ethyleneterephthalate) ,PET, is recovered from beverage bottles, which are mainly clear soft drinks . The depolymerisation of PET by glycolysis in an excess propylene glycol has been studied and the glycolyzed products are identified by chromatographic techniques. All products are hydroxyl terminated short chain segments and are used to produce unsaturated polyesters compatible with styrene. Fiber reinforced polymer (FRP) made with these resins is investigated and compared with those prepared using virgin resins .

In order to qualify the prepared FRP for use in LLW packaging, a materials testing program covering the physical properties, such as mechanical, thermal and durability properties, irradiation stability and microbiological response before and after environmental exposure was performed.

The properties of FRP using resins based on recycled PET are comparable to those obtained from FRP using virgin resins. The recycling of PET in FRP offer the possibility of a lower source cost of materials and would help alleviate an environmental problem and save energy.

*Symposium [J]
16:00-17:55, Jan.8*

Depolymerization Process in the Recycling of Nylons and Polyesters

Malcolm B. Polk

*School of Textile and Fiber Engineering
Georgia Institute of Technology, Atlanta GA 30332-0295*

The development of low temperature, atmospheric pressure processes for the depolymerization of PET, Nylon 6, Nylon 66 and Nylon 46 utilizing sodium hydroxide or hydrochloric acid and phase transfer catalysts will be described. A series of experiments were run in order to examine the applicability and efficiency of benzyltrimethylammonium bromide (BTEMB) as a phase transfer catalyst in the depolymerization of Nylon 6,6. A known amount of polymer fiber was mixed with 200 ml of a 50% aqueous solution of sodium hydroxide and 0.2g of BTEMB in 5 different runs. The mixture was heated in a mineral bath at a bath temperature of 1430°C and allowed to reflux for 24 hours with continuous stirring. Nylon 66 fibers were also depolymerized with 7% HCl solution in the presence of phase transfer agent at 800°C for 24 hours to produce adipic acid and HMDA-2 HCl in 85.3 and 70.9% yields, respectively. The blank run produced a 29.6% yield of adipic acid and a mixture of HMDA-2 HCl, oligomers and adipic acid.

Nylon 46 fibers were depolymerized with 50% NaOH and phase transfer agent for 24 hours at 1650°C to form a soluble product along with ca. 62% undissociated fibers. On the cooling of the solution, a powder settled to the bottom of the flask. The powder formed turned out to be a mixture of oligomers, adipic acid and 1,4-diaminobutane. Solid state polymerization was carried out on the nylon 46 oligomers at 2100°C for 16 hours. The viscosity average molecular weight increased from 1846 g/mole to 16,343. The melting point of the repolymerized Nylon 46 was 2800°C.

PET fibers were hydrolyzed with 5% NaOH @ 800°C in the presence of trioctyl methyl ammonium bromide. The reaction time was 60 min. and the yield of adipic acid was up to 93%.

*Symposium [K]
10:30-12:00, Jan. 9*

Use of Polymer Blends and Compatibilization Technology for Carpet And Textile Waste Recycling

Satish Kumar

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In 1995, approximately 8 billion lbs of carpet was produced in the USA, which consumed 3 billion lbs of nylon fiber. In the US, carpet is generally a four component composite system. Polypropylene is used for the primary and secondary backing, nylon 6 or 66 is the commonly used face fiber and CaCO_3 filled styrene butadiene rubber (SBR) is used as the adhesive joining the primary and the secondary backing. The face yarn is tufted into the primary backing. It is estimated that three or four billion lbs of new carpets production goes for replacing old carpets. Most of the used carpet currently goes to the landfills. Besides carpets, textiles, home and automotive furnishings are other sources of fibrous waste currently going to the landfills. The total fibrous waste, including textiles and carpet going to the US landfills, exceeds 8 billion lbs per year. Most of this waste is composed of nylon 6, nylon 66, and poly (ethylene terephthalate), with acrylics, polyurethanes, wool etc. forming the minor components.

Broadly speaking, fibrous waste recycling efforts are focused in the following areas (i) chemical processing, (ii) melt processing, (iii) composites manufacturing, and (iv) waste to energy conversion. In the area of chemical processing the emphasis has been on recovering chemicals either by depolymerization or by pyrolysis. Melt processing requires, either separation of individual components from the waste streams, or the use of compatibilizers to blend other, wise incompatible polymers and rubbers to achieve good mechanical properties. In the area of composites the following approaches have been taken. (a) use of fibrous waste with resins such as phenolics to make lumber replacement products, (b) to use fibrous waste for soil and concrete reinforcements, and (c) to use the fibrous waste thermoplastics as the matrix and a reinforcing fiber such as glass to make glass mat reinforced thermoplastics. The waste to energy conversion is not very popular in the US.

The various fibrous waste recycling approaches will be briefly reviewed. Melt processing and compatibilization study on carpet has been carried out. Compatibilizers such as polybond 3002 and Kraton rubber have been used. From his study, it has been demonstrated that compatibilization and blending is an effective method for recycling selected fibrous waste streams.

*Symposium [K]
10:30-12:00, Jan. 9*

Polyethylene/Polypropylene blends The Key of The Big Stream of Urban Solid Wastes. Chemical Modification Possibilities

E.P.Collar, J.Taranco, O.Laguna, J.Michelena, J.M.García-Martínez

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Following our first studies on polyolefin blends, this work shows the possibilities of chemical modification occurring in blends of polyethylene and polypropylene by the presence of a peroxide. This work was planned under the basis the more and more important role polypropylene is playing in the film fraction present in urban solid wastes. In these preliminary studies, polyethylene used for blending was LDPE (due to the fact the ratio LDPE/HDPE in urban solid wastes is 3.5/1). Both technical and economical difficulties of separation of both fractions (PP and LDPE) make the possibility of compatibilizing blend an attractive way to recover these materials.

In such a way, and looking for modifying the properties of the binary blend, the possibility of abstraction of the hydrogen from the tertiary carbon atom, which is supporting the methyl group in the repetition unit of polypropylene chain, appears to be a way to compatibilize the blend. This latter is the reason of using an initiator like peroxide for that purpose.

At this work we present preliminary results observed when blending PP and LDPE in a torque rheometer. The changes in torque values have been correlated to the gel fraction obtained from the blends and compared to those obtained for the virgin polymer.

Also thermal studies conducted under dynamical conditions were carried out and results are presented at this work.

*Symposium [L]
14:00-15:30, Jan. 9*

Zinc Ferrite Pigment for Corrosion Protection

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Zinc ferrite pigment was prepared by a solid-state reaction, A mixture of α - Fe_2O_3 and ZnO in a molar ratio of 1:1 was fired at 1200°C . X-ray diffraction measurements proved that the reacted material crystallized in aspinel structure. Measurements of the pigment specification and properties were carried out according to standard international methods. The pigment extracted and the paint of the pigment with linseed oil have been examined for protection of steel panels from rusting. The prepared pigment was incorporated in some paint formulations; physical, chemical and mechanical properties of the paint films were studied, they were also tested for corrosion resistance.

It was found that Zn-ferrite is a basic pigment, and can recommended for anti corrosive paints. Corrosion protection was evident for the formulated paints; protection increases as the pigment concentration increases. Protection mechanisms was followed up chemically and emphasized physically by the use of Mossbauer spectroscopy.

*Symposium [L]
14:00-15:30, Jan. 9*

Atactic Polypropylene. An Industrial Waste Useful as Interfacial Activity Modifier in Heterogeneous Materials Based on Polypropylene Chemical Reaction Process

J.M.Garefa-Martinez, O.Laguna, S.Areso, and E.P.Collar

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In this work, the results obtained in our studies on chemical modification of polyolefins with polar monomers by a radical process using initiators like peroxide are presented.

The possibilities of modifying atactic polypropylene, an industrial waste from polymerization process, is mainly enhanced. Such polyolefin let us obtain similar amount of grafted groups than other commercial products having a high price. The new products are used to improve interfacial activity in heterogeneous materials based on polypropylene (polymer blends and composite materials), in the same way, and having at least similar performance than commercial products. This study has been carried out by using Box-Wilson statistical experiment design for two, three, and even four independent variables as they are: initial concentration of peroxide and polar monomer, time of reaction and, in its case, gear rate.

Thus processes either in solution or in the molten state were modeled, and its results to be that reaction yielding was higher when the process was conducted on the molten state. The role played for the reaction time in order to maximize the level of grafting obtained is also clearly concluded.

As a consequence of these studies, we dispose of a broad range of modified polypropylenes containing different grafted groups: succinic anhydride, succinyl-fluoresceine, or p-phenylenebismaleamic acid; and with different grafting levels. These products proved to have a considerable capability as improver of interfacial activity when used in composite materials or polymer blends based on polypropylene.

*Symposium [L]
14:00-15:30, Jan. 9*

Business Session

**The Virtual Business:
A New Paradigm for Developing and
Commercializing Advanced Materials**

James Economy

University of Illinois at Urbana-Champaign, Urbana, IL

We have successfully prepared low cost activated carbon fibers by coating a glass fiber with a phenolic resin and then curing and activating the coating. Publishing of this work has generated widespread interest on the part of industry concerning their commercial use. On the other hand, there is very little interest on the part of industry in pursuing manufacture of these new fibers. This has led us to consider a new concept, namely establishing "a virtual business". The implications of this approach will be discussed.

*Symposium [B]
16:00-17:30, Jan.5*

New Business Opportunities for Lasers in Industry and Medicine

J.D. Bhawalkar, M. Casstevens
Laser Photonics Technology, Inc.
Amherst, NY

Recent development in laser technology have opened up new and practical opportunities in materials processing and medicine. At Laser Photonics Technology, we are working to develop laser systems which are highly cost effective solutions to such applications. This talk will highlight recent developments in laser technology as well as new applications to materials processing, medicine and surgery. Specifically, the talk will focus on diode lasers, diode-pumped solid-state lasers and compact CO₂ lasers. Applications for cutting, drilling, marking and engraving materials will be discussed. Medical applications will consist of low-level laser therapy, cosmetic and ophthalmic applications, as well as photodynamic cancer treatment.

Symposium [C]
10:30-12:30, Jan.6

The \$5000 World Car - A new Paradigm for The Twenty-First Century

Bill Jennings

President, Olympia Industries, Goleta, CA, USA

There is much talk in automotive industry of efforts to develop a "world car". By implication, the world car is one that would be appropriate for the vast populations of the world's developing countries as well as those of the developed ones. Such a vehicle would sell for approximately \$5000, be rugged enough to handle the poorest road conditions, and have a relatively large payload or passengers capacity. Until now, the world car has yet become a reality. Olympia Industries is about to introduce the Phoenix .. a true world car.

In one hundred plus year history, the automobile industry has experienced three significant changes in manufacturing systems. The earliest cars were hand built by skilled craftsmen who fabricated component part and then skilfully fitted each component part and then skilfully fitted each component together to complete one unique automobile. Even though cars built by one compony looked alike, no two were ever exactly the same. After the turn of the century, Mass Production introduced standardization in components and assembly techniques. This revolution in manufacturing improved quality and drastically reduced production costs making automobiles practical and affordable for the working class. The growing demand or improved quality, recognition of rapidly dwindling energy resources, and increased global competition within the last twenty years has given rise to the Just-in-Time/Quality approach to manufacturing. Each of these systems was appropriate for the economic times and conditions that dictated their development as well as for the markets they served.

All of these manufacturing techniques, however, have only a relatively small percentage of the world's population. They were developed in and for the wealthiest countries and, for the most part, have become impractical for the most of the smaller developing countries.

Olympia has created a new manufacturing technique which it calls Adaptive Manufacturing. Adaptive Manufacturing is a holistic approach to vehicle production that considers the practical needs of the consumer and the economic sector as well as the local resources in terms of available materials, work force, and technology.

This paper will discuss the development of the Phonix and the fundamental constructs of Adaptive Manufacturing which include vehicle design and material selection parameters to the application of appropriate technology in tooling, fabrication, and assembly.

*Symposium [C]
10:30-12:30, Jan. 6*

Surface Properties of Silicone High Voltage Insulators

Michael J. Owen

Dow Corning Corporation, Midland, MI USA

Polymeric insulator technology is a growing global opportunity with silicones offering one of the best combination of properties available. The chief advantages of polymeric insulators over conventional porcelain and glass insulators in outdoor high voltage (HV) insulation applications are light weight, superior vandal and environmental damage resistance, and better contamination performance. Polydimethylsiloxane (PDMS) is a polymer of higher thermal stability and lower surface energy than hydrocarbon polymers. It also has excellent weathering and ultraviolet radiation resistant properties as well as advantageous burning characteristics that make it particularly suitable for HV application when formulated into elastomers by the use of reinforcing silica fillers and the inclusion of heat dissipating fillers such as alumina trihydrate.

Polymers are more easily degraded than inert inorganic materials under exposure to discharges and arcing. Corona discharges and dry band arcing occur when the surface of an energized insulator is covered by an electrolytic film formed by the presence of moisture and contamination. The intense localized energy of the dry band arcs can cause material degradation in the form of tracking and erosion producing a wettable hydrophilic surface. It is a paramount requirement of a polymer in this HV application that its surface recover its hydrophobicity as rapidly as possible after such events.

The molecular architecture of PDMS is uniquely suited to beneficial hydrophobic recovery on exposure to corona discharge of sufficient intensity to damage the surface. The very low glass transition temperature and high free volume make the PDMS surface highly mobile and hydrophobic recovery mechanisms such as chain reorientation and diffusion occur readily. The presence of low molecular weight unreacted material in most commercial PDMS elastomers ensures a supply of such migratable species. Our studies of these effects using contact angle, scanning electron microscopy and X-ray photoelectron spectroscopy will be described.

*Symposium [I]
14:00-14:30, Jan.8*

Precision Polymer Technologies - A Xerox Licensing Business

Jim Sharp

Xerox Research Centre of Canada

Precision polymer technologies, a Xerox company, was formed in 1995 with a mission to license a unique polymerization process for the polymer industry. This process, known as VIVAMERTM technology, is based on Stable Free Radical Polymerization. The technical development of SFRP and the rationale for the establishment of PPT are reviewed. The potential polymer markets for licensing the technology are defined and several strategic models are discussed to exploit these markets. Using these markets and the predicted licensing, a long range plan is developed.

*Symposium [I]
14:00-15:30, Jan. 8*

Opportunities in Natural Fiber-Thermoplastic Composites

R. Rowell

USDA, Forest Products Library, Madison, WI USA

Combining agro-fibers (lignocellulosics) with other resources provides a strategy for producing advanced composite materials that take advantage of the properties of both types of resources. It allows the scientist to design materials based on end-use requirements within a framework of cost, availability, recyclability, energy use, and environmental considerations. Lignocellulosic resources have low densities, are low in cost, renewable, non-abrasive, have excellent specific mechanical properties, and are potentially outstanding reinforcing fillers in thermoplastic composites. The specific tensile and flexural moduli, for example, of 50% by volume of kenaf-PP composite compares favorably with a 40% by weight of glass fiber-PP injection molded composite. These new composite materials are finding new applications and new markets never before envisioned by the agro-based industry.

*Symposium [II]
14:00-15:30, Jan.8*

**Poster Sessions
I & II**

POSTER SESSION [I]

Electrical Properties of EL-AMRIA Pure PVC

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The rapid growth of PVC usage, due to its combination of properties, price and ease of processing, encourages continues research and development of PVC process, technology and compounding with different selected ingredients to improve its properties for wide range of applications. Electrical properties of PVC are of great interest because of their wide electrical applications, and which can considered as a one of the easily molded insulators. The electrical properties of PVC depends not only on chemical composition and structural features but also on the degree of molecular order. The dielectric investigations provide a fundamental method for studying the rotational and hopping of electrons processes involved in materials and account for the dispersion behaviour associated with molecular configuration and its ordering as affect the conductivity behaviour. In the present work the Egyptian brand with K value 67 from Al-Amria Petro-Chemical Co. is being studied. Dielectric properties and loss factors together with electrical conductivity at different temperature and frequency are given.

*Poster Session[I]
18:30-20:30, Jan. 6*

Electrical Conductance of Irradiated LDPE/NBR Conductive Blend During Swelling in Benzene

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The effect of γ -irradiation on both the electrical conductance and diffusion coefficient, D , of benzene in acrylonitrile butadiene rubber mixed with different concentration (1,3, and 5 phr) of low density polyethylene (LDPE) have been studied. The diffusion coefficient decreases with increasing γ -irradiation dose for loaded samples with 1 and 3 phr of LDPE content, while samples with 5 phr of LDPE show significant increases of diffusion coefficient with the increase of γ -radiation dose. The electrical conductance was found to be highly affected by γ -irradiation dose.

Electrical Properties of β -Eucryptite and β -Spodumene Ceramics

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Six compositions in the system Lithium alumina silicates (LAS) with different ceramic ratios of $\text{Li}_2\text{O}:\text{Al}_2\text{O}_3:n\text{SiO}_2$ where n ranged between 2 and 10 were prepared and vitrified using conventional techniques. The temperature dependence of dc conductivity (σ), dielectric constant (ϵ') and dielectric tangent loss angle ($\tan\delta$) were investigated. The data of electrical conductivity revealed that conduction mechanism is mainly ionic. Hopping mechanism between localized sites is assumed to be involved in process of charge carrier transport. The dielectric constant for β -eucryptite sample ($\text{Li}_2\text{O}:\text{Al}_2\text{O}_3:2\text{SiO}_2$) shows a unique behavior compared to β -eucryptite solid solution ($\text{Li}_2\text{O}:\text{Al}_2\text{O}_3:3\text{SiO}_2$) and β -spodumene sample ($\text{Li}_2\text{O}:\text{Al}_2\text{O}_3:4\text{SiO}_2$). It possessed one pronounced relaxation peak. Silica content was found to play an important role in the electrical properties of ceramic samples as ϵ' values are reduced in parallel with the enrichment of β -spodumene and quartz at the expense of β -eucryptite around 400k.

Emission Characteristics of a new Class of Strongly Fluorescent Phenanthrene Derivatives

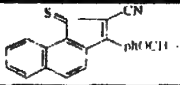
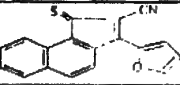
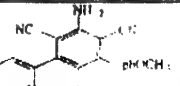
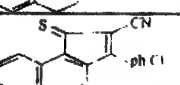
Nahed M.Fathy, Essam M.Hassan* and Khaled A.Abou-Zeid*

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* National Institute of Laser Enhanced Science (NILES), Cairo University, Egypt

Fluorescence quantum yield (Φ_f) of some highly fluorescent phenanthrene derivatives has been measured using diphenyl anthracene as reference in both ethanol and isopropanol, respectively

The critical transfer distances (R_0) between these compounds and rhodamine 6G, Distyryl pyrazine (DSP) and 1,4-Bis (β -Pyridyl-2-Vinyl) Benzene (P2VB) have been studied using steady state emission in isopropanol. The underlying mechanism of energy transfer is that of Förster type.

Compounds	Φ_f		R_0 Å		
	ethanol	isopropanol	R6G	DSP	P2VB
	0.99	0.98	50.4	57.6	41.9
	0.35	0.27	29.6	43.3	30.68
	0.60	0.43	49.7	45.1	33.3
	0.99	1.00	51.6	57.6	42.9

Environmental and Gamma-Irradiation Effects on The Optical and Electrical Properties of Modified Polyvinylchloride

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The effects of exposure of cadmium and lead-doped polyvinylchloride samples to both environment and gamma radiation were studied, using optical and electrical techniques. The optical measurements were performed in the 200-1100 nm wavelength range. The ac impedance spectroscopy measurements were carried out in air at room temperature in the 0.1-100 kHz frequency range. The optical results reveal linear rise in the optical band edge λ_g with the irradiation dose solely in the case of lead-doped PVC due to high shielding effect of lead. The optical energy band ΔE , on the other hand, decreases with both the irradiation dose and exposure time to environment. The results of the electrical measurements show a Debye mechanism of conduction. Such induced variations are discussed in terms of the structural shifts effected in the doped polymer.

Poster Session/II
18:30-20:30, Jan. 6

Spectroscopic and The Thermogravimetric Studies of Gamma Irradiated Poly(vinyl-alcohol) and Its CuCl₂-Doped Derivative

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Pure and doped (0.5, 1.0, and 1.5% CuCl₂) poly(vinylalcohol) PVA were gamma-irradiated in the dose range of 1-150 Mrad at room temperature. The optical and thermal features of the samples were spectrally and thermogravimetrically studied in order to find the effects of the irradiation dose and doping concentration. The absorption spectra were collected in the spectral range of 200-1100 nm, while the thermograms were measured in the temperature region of 300-900 K at a heating rate of 5 K min⁻¹. Thermally, remarkable changes in the decomposition temperature T_d with the doping concentration and in the activation energy with the irradiation dose were observed. Spectrally, the energy gap also was found to be sensitive to both the modifying effects of irradiation and dopant. This explains the dependence of the optical energy gap on the thermal T_d and E parameters.

*Poster Session[II]
18:30-20:30, Jan. 6*

Conjugated Polymer Microcavity Leds: From Device Engineering To Display Applications.

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Recently, it has been shown that conjugated polymers can be used as the emissive material in light emitting diodes (LEDs). The advantages of using this class of material come from their ease of processing, the ability to chemically 'tune' the emission colour, and their physical stability and mechanical flexibility. Devices have now been developed by electronics companies having lifetimes of thousands of hours, with market place products expected in the near future. Initial applications are envisaged as LCD backlights and monochrome displays. However development of full colour red-green-blue EL displays requires saturated emission colours from the individual pixel elements which is at present problematic using conjugated polymers. This is because their emission bandwidth is generally very broad due to inhomogeneous broadening and vibronic coupling.

Microcavities present a route to overcome this limitation. A microcavity is a Fabry Perot etalon, in which an emissive material (in this case a conjugated polymer) is placed between two mirrors separated by a few hundred nanometers. This results in a set of confined electromagnetic modes within the cavity to which the polymer emission can couple. It is demonstrated that coupling to these modes results in spectral narrowing and forward enhancement of emission. In addition, by controlling the optical mode wavelength within the cavity, the device emission colour can be varied. Using this means, EL display elements can be realised using conjugated polymers, having good spectral emission and high brightness. Results will concentrate on the technological application of polymer microcavities to displays, and in particular the fabrication of polymer based devices using standard photolithographic techniques. Various methods are presented to control the cavity mode wavelength, and sophisticated devices are presented where real-time spectral tuning of light-emission occurs.

The distribution of the confined field within the microcavity is investigated theoretically and experimentally. Structures are presented where a photoluminescent polymer layer is placed within a microcavity at a position corresponding to either a node or an antinode of the confined electromagnetic field. It is demonstrated that as expected, when the polymer is placed at an antinode of the confined field a forward enhancement of emission occurs. When it is placed at a field node, suppression of emission is seen. These structures optimise the coupling between the excited dipoles of the polymer and the confined electromagnetic-field, and show great promise for the development of low-threshold microcavity lasers.

Study of The Effect of Nd:YAG Laser on The Electrical Properties of Polycarbonate

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** National Institute of Lasers Enhanced Science, Cairo University, Cairo, Egypt*

The sample is a sheet of polycarbonate type A-bisphenol with thickness 3.5 mm. The dielectric constant and dielectric loss were measured as a function of temperature and frequency before and after irradiation with Nd:YAG laser (at green, UV, and IR regions).

A.C. electric conductivity and dielectric constant were measured for different temperatures and frequencies on unirradiated and irradiated samples of polycarbonates by Nd:YAG ($\lambda = 1.054, 532, 355$ nm). It was found that the a.c. conductivity of unirradiated and irradiated samples of polycarbonate were increased with increasing temperature and frequency. The activation energies for all samples at different temperature regions were calculated, it was 0.879 eV. The increase of the number of laser shots will decrease the conductivity and also the dielectric constant.

The threshold intensity for damage was determined. The possibility to find microcracks and microdefects was noted by using Scanning Electron Microscope (SEM) and Polarizing Reflection Optical Microscope (PROM).

*Poster Session[I]
18:30-20:30, Jan. 6*

Three Dimentional Data Storage

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Standard compact disks of WORM (Write Once Read Many) type are widely used for data storage now. Information is recorded on the disk surface, while the disk thickness is not used. The information capacity of disks can be enhanced by means of information recording in volume. Data storage in three-dimensional media was studied since 1960's, it is based on hologram multiplexing on the same location. Different optical schemes for hologram multiplexing at one location were proposed, namely angle multiplexing, wavelength multiplexing, phase coding of the reference beam, shift multiplexing, rotational and spatial multiplexing. Any of these methods or their combination can be implemented for the optical storage devices.

Holographic memory is usually associated with photorefractive crystals as well as with photochromatics, bacteriorhodopsin and other dynamic holographic media. Not disclaiming the importance of development of erasable materials we concentrate the efforts on stationery media that can permit to create the WORM type disks that have improved characteristics in comparison with the existing ones. We refer these materials as WORM materials. We chose this direction of investigation due to its wide perspectives in consumer market.

The review of WORM 3-D media developed in Russia that are suitable for the recording of thick holograms is done in the report. One of the main characteristics of these materials is their thickness, it should be about millimeter in order to reach the required selectivity properties of the hologram recorded. The manufacturing of such photosensitive materials meets a lot of difficulties; it is hard to produce these holograms with uniform properties (refractive index, transparency, sensitivity, etc.) and without reaction on postexposure development (absence of shrinkage, inhomogeneities in refractive index in depth, etc.)

The architecture of the optical scheme for hologram multiplexing depends on the choice of the photosensitive medium. The new scheme is proposed that can be implemented for the materials manufactured in the shape of optical fibers.

Electrical Properties of Gamma Irradiated Pure and Nickel Chloride-Doped Polyvinyl Alcohol Films

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The electrical transport properties, such as d.c. electrical conductivity (σ), dielectric constant (ϵ') and current-time characteristic of unirradiated and γ -irradiated pure and NiCl_2 doped PVA were studied in the temperature range 26-155°C. quantitative analysis was carried out to determine the activation energy, drift mobility and carrier concentration. The thermally activated mobility of charge carriers is confirmed from calculations of drift mobility at different γ -doses and temperatures. The results obtained revealed that γ -irradiation enhances the conductivity. The dielectric constant data at different temperatures before and after irradiation can be mainly attributed to the changes in the intra-and inter- molecular interactions.

The d.c. conductivity at 30 and 40°C, activation energy in low temperature region I and ϵ'_{max} for 20 wt% NiCl_2 proved to be dose dependent. The obtained data may allow to suggest that these materials may have an application in dosimetry.

*Poster Session[II]
18:30-20:30, Jan. 6*

Experimental Studies of Electronic Processes in Pi-Conjugated Polymer Films and Light Emitting Diodes (LEDs)

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Absorption, luminescence, photoinduced absorption, and optically detected magnetic resonance studies of electronic processes in pi-conjugated polymer films and LEDs will be reviewed and discussed. Particular attention will be given to the dynamic of singlet and triplet excitons, free trapped polarons and polaron pairs, and biopolarons. The implications for the performance of polymer LEDs, and possible relevance for small organic molecular LEDs will also be discussed.

*Poster Session[I]
18:30-20:30, Jan. 6*

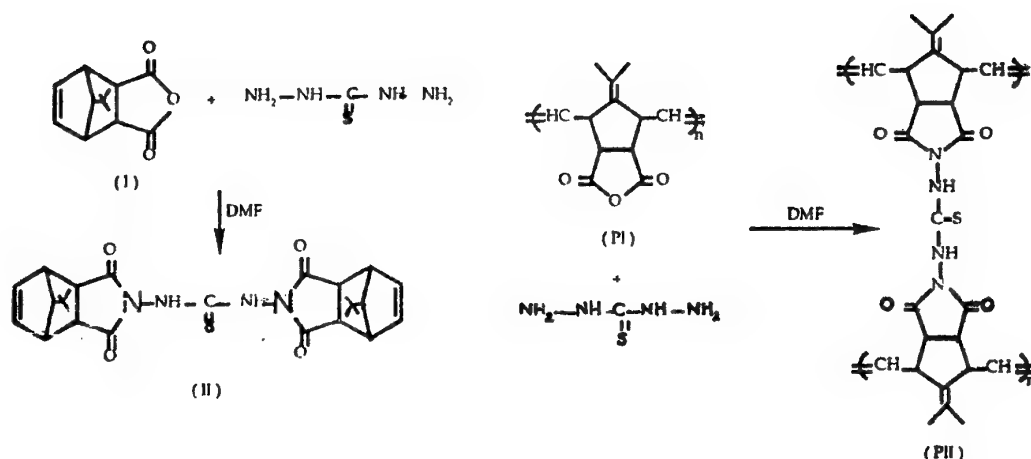
Synthesis, Comparative Studies of A.C. Conducting and Thermal Properties of Ring Opening Fulvene Polymer

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The work reported was to investigate the ring-opening metathesis polymerization of an imido-fulvene derivative monomer, using classical initiator, and to characterize the resulting monomer and network polymer expected, (the process is summarized in scheme 1). The a.c. conductivity of the monomer (II) and the polymer (PII) obtained were studied in the frequency range of 0.1 - 100 KHz, temperature interval 300 - 450 K. The Cole - Cole diagram have been used to determine the d.c. conductivity. The thermogravimetry and differential thermogravimetry were measured for both monomer and polymer in the temperature range 300-1000 K. Two endothermic peaks appeared in TGA Thermal curve. The estimated activation energy from the thermal analysis, is in a good agreement with that obtained from the temperature dependence of d.c. conductivity. Complexes derived from dimer (II) and polymer (PII) with $\text{Cu}(\text{Ac})_2\text{H}_2\text{O}$, $\text{Co}(\text{Ac})_2\cdot 4\text{H}_2\text{O}$ and $\text{Ni}(\text{Ac})_2\cdot 2\text{H}_2\text{O}$ were prepared and characterized by elemental analysis and optical spectroscopy. The thermal stabilities of monomer (II) and polymer (PII) and its metal complexes were studied by means of thermogravimetric analysis. The activation energies of the degradation of monomer (II) and polymer (PII) and its complexes were calculated.



Scheme (1)

Poster Session [I]
18:30-20:30, Jan. 6

Electroluminescent Polymer Devices: Charge Injection. Transport and Recombination

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One of the most exciting recent developments in polymer science is that of polymer electroluminescence. When a bipolar electrical current is passed through certain conjugated polymers they emit light. The intensity of emission is within the range required for practical displays and other lighting applications. By chemically altering the structure of the polymer backbone and sidegroups a whole selection of different colours across the visible spectrum can be produced. Ease of processing, the ability to cover large areas, mechanical flexibility, low cost, high efficiency and a low drive voltage make these materials very serious potential rivals for more established technologies. The development of conducting polymers raises the possibility of an all-plastic, large area, full colour display.

Polymer light emitting diodes (LEDs) consist of a transparent glass or plastic substrate, an optically transparent metal bottom electrode, a thin polymer film and a metal top electrode. To improve device performance it is important to understand how charge is injected from the metal electrodes, how it is transported through the polymer and how it recombines on the polymer chains to emit light. All of these factors can be directly related to device brightness, efficiency and drive voltage. A range of different experimental techniques were used to study these properties in single layer devices. Devices were prepared from poly(phenylene vinylene) (PPV) and three soluble derivatives PDAOPV, PmPV-co-DOctOPV and PPyV-co-DHepOPV. Current-voltage (IV) and current-luminosity (IL) characteristics and electroluminescent (EL) and photoluminescent (PL) spectra were recorded over a range of different temperatures. Thermally stimulated current (TSC), deep level transient spectroscopy (DLTS), optical DLTS (ODLTS) and admittance spectroscopy measurements were also conducted. Polymer film thickness and electrode material were also varied.

The IV characteristics show variations with temperature, the voltage required to produce a particular current increasing with decreasing temperature. Models involving only tunnelling injection are obviously inappropriate given the temperature variations. The increase in the voltage required to produce a particular current with increasing polymer film thickness also shows that, unlike traditional inorganic semiconductor materials, any model cannot involve injection alone but must include bulk transport. These characteristics will therefore be discussed and analysed in terms of combined models involving space charge and trap limited bulk transport and thermionic emission and tunnelling injection. TSC, DLTS, ODLTS and admittance spectroscopy results give direct evidence of the role of traps in the characteristics. The

IL characteristics and EL spectrum show that the luminosity for a given current increases with decreasing temperature. The increase in PL efficiency with decreasing temperature can only account for a fraction of the increase in EL efficiency. This can be analysed in terms of the decrease in the mobility of the carriers increasing the recombination capture rate, decreasing the number of carriers transmitted or recombined at deep traps and decreasing the level of quenching by moving the recombination zone away from the cathode. These factors can be directly related to the IV characteristic models. The EL and PL spectra both redshift with decreasing temperature and the oscillator strength is redistributed between the vibronic modes. The redshift can be related to both the increase in polarisation interactions as the interchain distances decrease and the increase in conjugation as the torsional vibrational modes which effect backbone planarity are frozen out and the backbone undergoes thermal contraction.

Odd-Even Oscillations of SHG Efficiencies in Dimeric Liquid Crystals

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Indian Institute of Science, Bangalore 560012, India*

Dimeric liquid crystals (DLC) have been extensively investigated as low molar mass models of polymeric liquid crystals in which the mesogens are linked together by flexible spacers to form the polymer backbone. In such polymers a significant oscillation of the isotropization temperatures (T_i) and also the isotropization entropies (ΔS_i) have been observed. Monomeric liquid crystals, in which the mesogen is linked to a parafinic tail, on either of both ends, also exhibit such odd-even oscillations but to a much smaller degree. In DLO's in which two mesogens are linked by a flexible polymethylene spacer in the sequence, mesogen-spacer-mesogen, these oscillations are more pronounced and often fall between those observed in polymers and monomers. Hence, such dimmers have often been considered as ideal models in the conceptual progression, monomer-dimmer-polymer.

Dimmer liquid crystals having two azobenzene mesogens linked by a flexible polymethylene spacer of varying lengths were synthesized. A strong electron accepting nitro group and an electron donating alkoxy linkage incorporated into these dimmers, make them also potential candidates for observing 2nd order nonlinear optical (NLO) activity. Most of the dimmers were shown to exhibit a nematic mesophase and the isotropization temperatures did exhibit the expected odd-even oscillations. Furthermore, it is noticed that the second harmonic generation (SHG) efficiencies, $I_{2\omega}(\text{sample})/I_{2\omega}(\text{urea})$ [in powder form], of the dimmers also exhibited these odd-even oscillations. These values for the dimmers belonging to the odd series (ranged between 1.00 and 0.019) were found to be significantly larger than those belonging to the even one (ranged between 8×10^{-2} to 2.1×10^{-2}). Furthermore, both in the even and odd series these values decrease with the increase in the spacer segment length, the decrease being more pronounced at small spacer length. However, at longer spacer lengths ($n > 4$) the variation within both the odd and even series is rather small. This is probably due to the fact that the spatial correlation between the two chromophores would decrease with increasing value of "n". The significantly higher SHG efficiencies for the odd series implies that there is a less effective cancellation of dipoles associated with the chromophores in odd dimmers as compared to other even ones.

Synthesis and Liquid-Crystalline Properties of Cholesteryl Esters from Philippine Coconut Oil

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In an effort to synthesize liquid crystals from locally available materials, cholesteryl esters were prepared from the reaction of cholesterol and fatty acids obtained from coconut oil in good yield. The prepared esters were found to exhibit both the smectic and nematic textures as observed with the polarizing microscope. Several formulations using this material were studied in terms of potential thermochromic and electro-optic applications. The synthetic route to this coco-cholesteryl esters and the mesomorphic properties of various formulations will be presented during the talk.

*Poster Session[II]
18:30-20:30, Jan. 6*

Commercial Development of Advanced Materials Technology

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Foster-Miller has been actively involved in the development of advanced materials technology and transferring the technology to the commercial market place. This paper will discuss several approaches being followed for commercialization including licensing, strategic partnerships and new spin-out companies to manufacture products. Case histories will cover thermotropic Liquid Crystal Polymers, Z-direction reinforcement of composites, and switchable holograms using polymer dispersed liquid crystal. The paper will conclude with an identification of business opportunities for collaboration with interested parties.

Thermodynamic of Sr-Mg Vermiculite Exchange and the Effect of PVA on Mg Release

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** National Research Center, Cairo.*

The aim of this work is to study thermodynamics of Sr-Mg vermiculite exchange and the effect of polyvinyl alcohol (PVA) on the interlayer magnesium release from the clay structure. The results indicated that the affinity of adsorption decreases as the unhydrated ionic radius increases.

Also it was found that the surface of vermiculite exhibits more selectivity for Sr by treating Mg-vermiculite with PVA than in its absence. With increasing temperature the exchange of Mg by Sr increases. The enthalpy value (ΔH) was positive, i.e. the exchange reaction is endothermic.

X-ray diffraction of the clay indicates that the C-spacing of vermiculite increases gradually as the adsorbed amount of Sr ion increases on the vermiculite surface.

The activity coefficient ratio of f_{Sr}/f_{Mg} varies at 20-C from 42 at 10% Sr saturation to 364 at 90% Sr saturation, therefore the ratio increased with the increase of Sr saturation.

*Poster Session [II]
18:30-20:30, Jan. 6*

Flocculation and Coagulation of Ca- and K-Saturated Montmorillonite in The Presence of Poly ethylene Oxide

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The objective of this work is to study flocculation of Ca-and K-montmorillonite in the presence of coagulant (CaCl_2 and KCl and a mixture of coagulants and polyethylene oxide M.wt.600,000) through measuring zeta potential. The action of Ca^{++} was more effective than K^+ decreasing the zeta potential of Ca-montmorillonite, consequently the electrical double layer of the clay particles has been reduced. On the other hand the action of K^+ was more pronounced than Ca^{++} in decreasing the zeta potential Of K-montmorillonite. This contradicting result was attributed to the change of the partial molal volume of Ca- and K-montmorillonite.

The flocculation of Ca-And K-montmorillonite by polyethylene oxide was enhanced when the clay was initially treated with electrolyte. The electrolytes reduces the thickness of the electrical double layer. Under this condition, the polymer segments collapse on the clay surfaces leaving polymer loops and tails available for contacting adjacent clay particles.

*Poster Session[II]
18:30-20:30, Jan. 6*

On The Change of Optical Energy Gap of Polyoxymethylene Compacts

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The effects of γ -radiation on the optical properties of polyoxymethylene (POM) compacts are studied at room temperature. The UV-visible spectrometer is used to study the optical absorbency in the wavelength range 200 - 1100 nm. The optical energy for both direct and indirect transitions as well as the energy band tail ΔE are determined as function of γ -exposure doses. The obtained results showed that the POM compacts became brittle as the γ -radiation reaches 5 M Rad. in the meanwhile λ_g increased to a maximum of 2 M Rad and the energy band tail ΔE reaches 5 eV at the same dose. Both direct and indirect energies of transition decrease with the irradiation dose. The results can be explained on the bases on γ -irradiation induced changes due to cross linking in the POM.

*Poster Session [II]
18:30-20:30, Jan. 6*

POSTER SESSION [II]

**Effects of The Electronic Field and Gamma
Radiation on The Optical Properties
of Modified Low Density Polyethylene**

M.E. Kassem, M. El-Muraikhi, H. El-Medfa, R.R. Amin and H. Mahmoud
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The effects of electric field and gamma radiation on the optical properties of low density polyethylene LDPE grade CD 0230 have been studied. The samples were irradiated at gamma doses of 0-100 Mrad and varying electrical field of $0-10^4$ V/cm. The optical absorption spectra were measured in the wavelength range of 200-1100 nm. The absorption coefficient E_{opt} and the energy gap ΔE values were found to be gamma dose dependent. Also, these optical parameters show a parabolic dependence on the electric field. The change in the polymer crystallinity due to irradiation can be attributed to the induced bond scission and subsequent cross linking.

*Poster Session [II]
18:30-20:30, Jan. 8*

Production of Poly(3-Hydroxybutyrate) by High Cell Density Fed-Batch Culture of *Alcaligenes Eutrophus* With Phosphate Limitation

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High cell density fed-batch fermentation of *alcaligenes eutrophus* was carried out for the production of poly(3-hydroxybutyrate), PHB, in a 60 L fermentor. During the fermentation, pH was controlled with NH_4OH solution and PHB accumulation was induced by phosphate limited instead of nitrogen limitation. The glucose feeding was controlled by monitoring dissolved oxygen concentration in the culture broth, which is a DO-stat method. The glucose concentration fluctuated within the range of 0 and 20 g/L. We investigated the effect of initial phosphate concentration on the PHB production when the initial volume was fixed. Using an initial phosphate concentration of 5.5 g/L, the fed-batch fermentation resulted in a final cell concentration of 281 g/L, a PHB concentration of 232 g/L, and PHB productivity of 3.14 g/L.h, which are the highest values ever reported to date. PHB content, cell yield from glucose, and PHB yield from glucose were 80% (w/w), 0.46 and 0.38, respectively.

*Poster Session [II]
18:30-20:30, Jan. 8*

Dynamic Mechanical Relaxation Behavior of SBS and SBR Copolymers and Their Blends

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Dynamic mechanical measurements have been carried out for blends of styrene-butadiene-styrene, SBS, (block copolymer) and styrene-butadiene rubber (random copolymer) with different composition ratios, namely 0,25,50,75 and 100%. The shear compliance, j' & j'' , were measured at various frequencies ranging from 0.01-30Hz over a wide range of temperature (-70- 150°C). The blockcopolymer, SBS shows two different glass relaxation process for each component, one appearing at (-60°C) while the other appeared at (70.5°C) corresponding to butadiene and polystyrene phases respectively. The exhibition of two independent glass processes implies that in spite of the covalent bonds linking the different blocks, styrene and butadiene, each component of the block copolymer forms a separate phase. Each phase has its own dynamics and can relax independently of the other. On the other hand, the shear compliance measurements of the blends of SBS and SBR with different styrene contents (23% and 40%) show that the glass process of either the butadiene phase or both of butadiene and styrene phases of SBS are shifted on the frequency axis and therefore indicating compatibility. Accordingly, it is concluded that blends.SBR (with 23% styrene content) is compatible with butadiene phase of the SBS, while SBR(with 40% styrene content) shows compatibility with both phases (styrene and butadiene phases).

Furthermore, Master curves for each process of the different phase have been constructed indicating some interesting flow and viscoelastic behavior. In addition DSC measurements have been carried out.

Poster Session [II]
18:30-20:30, Jan. 8

Dielectric Investigation of Crystallization Process of Polyhydroxybutyrate

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Polyhydroxybutyrate, PHB, is a naturally occurring polymer that can be produced and degraded by bacteria. PHB has some interesting properties which could qualify it as a substitution of many of the known synthetic polymers. It is a highly crystalline thermoplastic polymer, which has good mechanical and barrier properties. However, the technical use of this material is limited by two major problems, namely; the difficulty of processing as well as the aging of the material. The earlier problem arises from the fact that the PHB crystals melt first at temperatures around 180°C and degrade very rapidly at temperature greater than 185°C.

On the other hand, the aging process is attributed to progressive crystallization process that taking place around room temperature (storage) and causes a great loss in mechanical properties and cracks in the product. For this reason, the present work aims at investigating the crystallization process on the lowest molecular level (dipolar segments) which represents the elementary unit required for the crystal growth by using the dielectric method.

Several types of measurements are applied, namely:

1. Temperature scan at constant heating rate at various frequencies.
2. Temperature scan at different heating rates at constant frequency (10kHz).
3. Isothermal measurements over extended period of time at various temperatures namely; 10, 20, 25, 30, 40, 110, 130, 150 °C.
4. Cooling curves from the melt with a different rates.

The results obtained showed that:

1. The optimum cold crystallization process is about 25 °C.
2. The onset crystallization time, is temperature dependent and can be reduced to be less than 2 min. at 40 °C.
3. The equilibrium crystallization time can be attained after more than one hour at 30°C.
4. Crystallization from melt takes place in three steps at three different temperatures. Furthermore, it was possible to evaluate the temperature, and the percentage of crystallization for each step. In addition, it is possible to describe the obtained results theoretically.

*Poster Session [II]
18:30-20:30, Jan. 8*

Control of Thermal Elimination Temperatures of Poly(Phenylene vinylene) Precursors

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Poly(phenylene vinylene) and its derivatives have been mostly synthesized via the Wessling route wherein a sulfonium polyelectrolyte precursor is thermally eliminated to give the final conjugated polymer. Treating the sulfonium precursor with methanol or even carrying out the polymerization directly in methanol was shown to be an effective method for generating an organic soluble methoxy precursor. This allowed the complete soluble methoxy precursor. This allowed the complete solution characterization of the precursor polymer and further made the preparation of films and fibers of PPV and its derivatives a lot easier. However, while the elimination of the sulfonium precursor can be effected at 220-250°C, the elimination of the methoxy precursor happens at these temperatures only in the presence of an external acid, typically under a slow purge of HCl gas. Thus, while the processing was rendered a lot easier, the elimination process was made more cumbersome. In an attempt to control the elimination conditions and temperatures, we have been interested in developing alternate organic soluble PPV precursors by reacting the sulphonium precursors with nucleophiles other than methanol.

We report here the synthesis of poly(2,5-dimethoxyphenylene vinylene), DMPPV, precursors that have two eliminatable groups, which are prepared by competitive nucleophilic substitution of the sulfonium precursor with methanol and water. The resulting precursor copolymer contained both hydroxy and methoxy groups in various ratios depending upon the starting solvent composition. The hydroxyl groups were further converted to more readily eliminatable acetates by treatment with acetyl chloride and pyridine. The structures of all these copolymers were characterized by ¹H-NMR spectroscopy and their thermal elimination characteristics were examined by TGA. The structures of the eliminated conjugated polymers were confirmed by IR and UV-visible spectroscopies.

*Poster Session [II]
18:30-20:30, Jan. 8*

Role of Solvent in Thermal Imidization of Polyamic Acid

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Imidization is known to proceed faster in the presence of amide solvents. The widely accepted explanation for this behavior is that the solvent molecules allow the reacting species to attain favorable conformations for imidization. However, it is also possible that the solvent could aid the reaction by serving as a proton acceptor-donor, and assist in the loss of carboxyl proton. The solvent molecule could function as a proton sink, accepting the proton from the carboxyl group and then donating it back after ring closer. Amide solvents such as N-methyl pyrrolidinone are known to form strongly hydrogen bonded complexes with carboxylic acid. Thus the transfer of a proton to the basic solvent under the imidization conditions would seem quite likely. In this paper, the kinetic study on thermal imidization of the solvent-free polyamic acid, 4,4'-oxydiphenylene pyromellitic acid, is reported. By investigating imidization behavior in the absence of solvent and comparing it with that in the presence of solvent, we could deduce the role of solvent on imidization. Temperature-variable FTIR spectroscopic technique was used to monitor time-dependent changes of degree of imidization. To quantitatively analyze the results, cage model was applied to the system and the various physical parameters for imidization of the solvent-free polyamic acid were determined.

As increasing reaction temperature, initial increasing rate of degree of imidization and eventual degree of imidization increased. The effective rate constant was found to be significantly lowered at the initial stage of imidization reaction due to the rapid decrease in chain mobility. With the cage model, the amount of the amic acid unit which can attain favorable conformation for imidization before kinetic stop is reached, and the true chemical reaction constant were determined. Both the amount of the amic acid unit and the true chemical reaction constant increased with temperature. From the temperature dependency of chemical reaction constant, it was found that the activation energy of thermal imidization in the absence of solvent is 25.0kcal/mol, which is consistent with the one determined for the solvent-containing polyamic acid. This result indicates that the solvent does not take part in imidization reaction via a direct way such as proton transfer, but only plasticizes the reacting medium aiding the transformation of amic acid unit to the conformation favorable for imidization.

*Poster Session [III]
18:30-20:30, Jan. 8*

Polymerization and Copolymerization Behavior of N-Acryloyl,N'-Caynoacetohydrazide

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Radical polymerization and copolymerization of N-acryloyl, N'-cynoacetohydrazide (ACAH) were carried out in DMF in the presence of azobisisobutyronitrile (AIBN) as an initiator. The homopolymerization behavior of ACAH was investigated kinetically. The stability constant of ACAH and PACAH complexes with Cu^{II} and Fe^{II} cations were calculated. The rate of copolymerization of ACAH with different conventional monomers as vinyl acetate (VA), methyl methacrylate (MMA) and acrylonitrile (AN) was measured as a function of the mole fraction of comonomers. The reactivity ratios (r_1, r_2), Q and e values for ACAH were determined. The thermal stability of the prepared polymer and its copolymer with An was investigated using TGA.

*Poster Session [II]
18:30-20:30, Jan. 8*

Structure-Properties Relationships of Graft And Block Copolymers

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Graft and block copolymerization reactions are important ways of polymer, fiber and textiles modification. Recently graft and block copolymers are widely used as compatibilizer in polymer and textile coating and adhesion. The properties of the systems depend not only on the chemical nature of the components of the copolymers but also on its composition and physical structure. Major effects on the complex properties are caused by the nature of grafting reaction whether it is homogenous or heterogeneous, the number and length of the grafts per molecule of the substrate, the length and alternation of the blocks, crystallinity and morphology of the components. Quantitative evaluation of the effect on the bulk properties of the modified macromolecules whether they are fibers, films or blocks, in addition to the chemical nature of the substrates and the grafts, is discussed and given in this review.

*Poster Session [II]
18:30-20:30, Jan. 8*

Chemicals, Polymers and Ceramics from The Beach-Fourth Generation

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The search for new polymeric materials that meet or exceed the physical properties of commercial, carbon based polymers must eventually consider inorganic and organometallic materials. However, unlike carbon based polymers which rely on petroleum or natural product feedstocks; potential feedstock chemicals for non-carbon polymers are not readily available.

This presentation will describe methods of making rheologically useful trimetallic alkoxides and related polymers directly from silica and aluminum hydroxide and group I or II metal hydroxides/oxides, by reaction with ethylene glycol, triethanolamine and base. These compounds provide the low-temperature processability of organic polymers; yet convert on heating, to a wide variety of aluminosilicate ceramic materials. Thus, they provide access to many types of ceramic shapes including; ultrafine powders, coatings and fibers. Nanopowder powder processing will be emphasized in this presentation.

*Poster Session [II]
18:30-20:30, Jan. 4*

Simulations on filler Reinforcement in Elastomers

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Monte Carlo (MC) computer simulations based on the rotational isomeric states (RIS) model are used to predict the dependence of the modulus of filled polymer networks on the excluded volume of the filler particles and non-Gaussian characteristics of the chains. The filled polymer network is modeled as a composite of the cross-linked polymer chains and the filler particles. The numerical calculations cover the effects of network chain length, filler particle size, and size of the unit cell of the filler lattice. The present theoretical approach includes simple model calculations in order to examine possible molecular mechanisms of the reinforcement.

Our studies had been concerned with networks where poly(ethylene) (PE) and poly(dimethylsiloxane) (PDMS) chains were attached at one end to spherical filler particles. These studies had been extended by considering the elastic properties of PE and PDMS networks filled by spherical, ellipsoidal, and prolate particulate fillers placed on a three-dimensional cubic lattice. The work has currently been extended to study the reinforcement of PE and PDMS by randomly distributed filler particles. Also, modeling the complex phenomena of adsorption of elastomeric chains onto the filler surface has been carried out.

As would be expected, the results thus obtained underscore that both reinforcement and induced anisotropy of the network distribution functions as well as the stress-strain isotherms are affected by the size, shape, orientation of the filler particles, and the volume fraction of the filler particles in the network. In addition, the filler particles are found to increase the non-Gaussian behavior of the chains.

*Poster Session [II]
18:30-20:30, Jan. 8*

Lignin Filled Composites

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Lignin, a polyphenolic and polymeric material acting as a binder to the cellulosic fiber in wood is produced as a by-product in paper industry and its disposal is a serious environmental problem. If it can be used as a filler in plastics, it could consume large volumes and commercially this can be attractive. Due to the phenolic nature, lignin can react with formaldehyde to produce phenolic resins. However, the properties of such resins have been found to be inferior. Therefore, partial replacement of phenol in synthesis of phenolic resins and of cellulosic fillers in moulding powder formulations may be important. The present work looks at (i) properties of thermoplastic composites using lignin as a filler, (ii) preparation and characterization of novolak resins made from mixture of (lignin/phenol) and properties of corresponding moulding powder and (iii) properties of conventional moulding powders using lignin as a filler in place of wood flour.

Dry lignin in the powder form was blended with commercial grade polypropylene (PP), low density polyethylene (LDPE) and high density polyethylene (HDPE) upto 30% by weight, using HAAKE RHEOMIX 600 employing a pair of counter rotating universal rotors. Mechanical, electrical and rheological properties of the resulting composites were measured. Effect of ethylene acrylic acid copolymer, EM and coupling agent (Titanate) on these properties was also evaluated. Phenol formaldehyde novolak resins were prepared from mixtures of (phenol + lignin) by varying lignin contents upto 30% by weight. The resulting resin was analysed for its properties and moulding powders were prepared using resin 42%, wood flour 25.3%, coconut shell powder 19.4%, hexamethylene tetramine 6.3% and additives 7%. Mechanical, electrical and flow properties were evaluated. In another set of experiments, a commercial grade resin was compounded into a moulding powder but gradually replacing the wood flour by lignin. The change in properties was evaluated.

The tensile strength of lignin filled thermoplastic composites steadily decreased and melt viscosity increased as amount of lignin increased, but storage modulus remained relatively unaffected. The gelling times of resins remained unaffected. The flow properties indicated higher viscosity. The tensile strength decreased by 28% due to incorporation of lignin (30%). The electrical resistance, however, increased by a factor of 1.91. When lignin was used in place of wood flour, the decrease in tensile strength was much more (76%) but electrical resistance increased by a factor of 4. This indicates that lignin may not be compatible with phenolic resin in moulding powders. Use of proper compatibilizer may be worthwhile to study.

*Poster Session [III]
18:30-20:30, Jan. 8*

Wear Behavior of Copper-Iron Composite Material

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A composite was made by still casting from copper matrix and chips of plain carbon steel as reinforcement. The steel chips were heated to 910°C prior to the casting process. The composite was characterized and showed good mechanical and thermal properties.

This study examined the structure, processing and, to a great extent, the wear behavior of this composite. It was found that wear depends mainly upon the chips and their volume fraction.

*Poster Session [II]
18:30-20:30, Jan. 8*

First Man-Made Composite Material

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It is well acknowledged in the field of composite materials that the first composite was made by the ancient Egyptians. This is the burnt Brick that is still made in many parts of Egypt. The manufacturing procedures used are still the same as they were in ancient Egypt. The Nile mud is first moistened, mixed thoroughly with short pieces of wheat straws. The mixture is afterwards next placed in wooden molds, then placed in the sun for few days to dry. Following that, the dried bricks are arranged in gamins that acts like a kiln and the burning process leads to chemical reactions and voids formation in place of straws. The resultant product is the first man-made composite material in history. In service, the voids arrest and bunt the crack propagation in the bricks which suggests the awareness of the ancient Egyptians of failure mechanics. In this study, the composite is reproduced, characterize, and its properties were compared with some of the bricks that are used in Egypt and elsewhere. Furthermore, the study examines the overturning process on the different properties of the composite.

*Poster Session [II]
18:30-20:30, Jan. 8*

Recovery of Some Metal Ions Using Gamma Radiation Induced-Grafted Waste Rubber Powder

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Waste rubber powder has been grafted with different vinyl monomers by gamma radiation. The efficiency of the products obtained for some metal ion recovery was evaluated. Besides, their efficiency for the recovery of phenol as organic pollutant was studied. the monomers used for grafting are acrylic acid, acrylamide and acrylonitrile. Furthermore, the efficiency of the sodium salt of grafted polyacrylic acid as well as the amidoxime derivative of grafted polyacrylonitrile onto waste rubber powder was evaluated. It was found that the grafted waste rubber powder is able to recover lead, copper, cobalt and Nickel ions from their corresponding salts depending on the type of monomers used. Also, It was found that phenol can be reasonably recovered by using powder grafted with acrylamide.

*Poster Session [III]
18:30-20:30, Jan. 8*

State of The Art And Emerging Trends in Vehicle Recycling

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The technology of vehicle recycling today is capable of recovering and reusing about 75% of the weight of scrap vehicles. The 75% recycling rate is believed to be the highest recycling rate in durable goods. Moreover, with a technology that was developed in the '70s, vehicle recycling is a profitable undertaking and, consequently, a robust infra-structure for handling and recycling scrap cars exists today. The recovery of materials from scrap vehicles, however, is generally limited to metals, such as steel, iron, zinc, aluminum, and copper. Non-metallic materials are usually discarded to landfills. The technology of recycling plastics and other non-metallic materials, including fluids, from vehicles is at its infancy and its profitability is yet to be determined. Several trends are developing in the automotive industry which will have a positive effect on recycling. Examples are the recent applications of recycled plastics, elastomers and fluids, the marking of plastic parts, and the joint R&D recycling projects between the automotive companies, suppliers, and recyclers.

*Poster Session [III]
18:30-20:30, Jan. 8*

N-Acryloyl, N'-Cyanoacetohydrazide as Thermal Stabilizers for Rigid Poly (Vinyl Chloride)

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N-acryloyl, N'-cyanoacetohydrazide (ACAH) and its complexes with some metal ions have been investigated as thermal stabilizers for rigid poly (vinyl chloride) (PVC). The stabilizing efficiency of ACAH and its complexes has been measured by the continuous potentiometric determination of the evolved HCl gas resulted from the degradation process and by the extent of discoloration of the degraded samples as compared with conventional thermal stabilizers. The efficiency of blending ACAH stabilizers has been also investigated. A probable mechanism for the stabilizing action of ACAH has been also proposed.

*Poster Session [II]
18:30-20:30, Jan. 8*

Thermal Stability of Acryloylbenzoic hydrazide Homopolymer and Its Complexes With Some Transition Metals

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Polycryloylbenzoic hydrazide and its complexes with some transition metal were prepared and characterized by elemental analysis, IR, UV-V16 Spectra and magnetic measurements. The electrical properties and thermal stability of the polymers were investigated. The thermal stability of the polymer complexes are higher than the homopolymer. The electrical measurements indicate that the homopolymer and its metal complexes are within the insulator range.

Tailoring Push-Pull Polyenes and Related Systems with Large Nonlinear Optic Responses

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Polyenes with substituted electronic push and pull groups such as amino and nitro groups besides breaking the inversion symmetry of the system also leads to large changes in the dipole moment between the ground and excited states. Hence these systems are expected to show very large response to second harmonic generation (SHG). These systems are modeled excited states. Hence these systems are expected to show very large response to second harmonic generation (SHG). These systems are modeled employing the Pariser-Parr-Pople (PPP) Hamiltonian and the model exact SHG coefficients are obtained using a correction vector scheme. We note that the two state model (TSM) of Oudar and Chemla predicts SHG coefficients which are larger than the model exact values. There is no systematic trend in the quantitative deviation of TSM predictions from the exact results. The exponent for the size-dependence of the SHG coefficients depends strongly on the magnitude of the push and pull strengths. The SHG coefficient also shows very increase as the central double bond is twisted close to 90° , although when the conjugation is broken (twist angle = 90°) the SHG response drops suddenly. In polysubstituted push-pull systems, the SHG response is maximum for the push-pull groups located on opposite sides of the chain. Here again twist in the geometry enhances SHG coefficient for $\theta \approx 70^\circ$. The studies are extended to the third harmonic generation (THG) response in real polymeric system such as the cyanine dyes, azacyanine dyes and polymethinimines. We find that long chain cyanine dyes exhibit the largest THG response in this group. We also discuss recent experimental and theoretical studies of SHG coefficients of the acidic and exhibit the largest THG response in this group. We also discuss recent experimental and theoretical studies of SHG coefficients of the acidic and basic forms of weak organic acids.

*Poster Session [II]
18:30-20:30, Jan. 8*

Effect of Heat Treatment and pH on the Corrosion Behavior of Ni-P Amorphous Alloys

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Ni-P amorphous alloys having different P content were prepared by electrochemical deposition. The amorphous structure of as-deposited films was characterized using thermal analysis and XRD techniques. The corrosion behavior of as-deposited and heat treated samples was studied in an acidic and neutral medium using potentiodynamic polarization technique. The protectiveness offered by the amorphous films against corrosion was also measured as a function of pH.

The results indicate a high compositional dependence of thermal and corrosion behavior of the amorphous alloys. Such behavior was found to be also sensitive to the existence of microcrystallites within the amorphous matrix.

*Poster Session [II]
18:30-20:30, Jan. 8*

Electrical Conductivity Studies on Impregnated Paper Sheets with Egyptian Eichhornia Crassipes Phenol Polycondensate Resins.

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Electrical conductivity studies on impregnated paper sheets with some Egyptian Eichhornia crassipes phenolic resins modified with phenol formaldehyde and phenol benzaldehyde were carried out. The insulating properties were found to increase in the following order: paper without impregnation, paper impregnated with modified resin with phenol benzaldehyde, paper impregnated with Eichhornia Crassipes phenol resin then paper impregnated with modified resin with phenol formaldehyde. The insulating properties of paper sheet is improved by the impregnation with the prepared resins. The d.c. electrical conductivity was measured from room temperature up to 100 °C. The mechanism of conduction was interpreted and explained in terms of the current theories.

*Poster Session [II]
18:30-20:30, Jan. 8*

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